AMERICAN SOCIETY
FOR
TESTING MATERIALS

ORGANIZED IN 1898
INCORPORATED IN 1902

PROCEEDINGS
OF THE
TWENTY-EIGHTH ANNUAL MEETING

Held at Atlantic City, New Jersey
June 23-26, 1925

VOLUME 25

PART I. COMMITTEE REPORTS
NEW AND REVISED TENTATIVE STANDARDS
LIST OF STANDARDS AND TENTATIVE STANDARDS

PUBLISHED BY
AMERICAN SOCIETY FOR TESTING MATERIALS
1315 Spruce Street, Philadelphia, Pa.
1925

Entered according to Act of Congress by the
American Society for Testing Materials,
in the office of the Librarian of Congress, at Washington.

Printed in Philadelphia, U. S. A.

The Society is not responsible, as a body, for the statements and opinions
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CONTENTS

PROCEEDINGS

PART I

PAGE

Summary of the Proceedings of the Twenty-eighth Annual Meeting........... 11
Promotion of Knowledge of Engineering Materials: Annual Address by the President—F. M. Farmer.. 34
Annual Report of the Executive Committee.............................. 46

COMMITTEE REPORTS

Ferrous Metals

Report of Committee A-1 on Steel.. 70
  Appendix. Proposed Revisions in Standards for Steel.................. 75
Report of Committee A-2 on Wrought Iron.................................. 77
Report of Committee A-3 on Cast Iron..................................... 84
  Discussion.. 87
Report of Committee A-4 on Heat Treatment of Iron and Steel.............. 94
Report of Committee A-5 on Corrosion of Iron and Steel................... 95
  Report of Sub-Committee III on Inspection of the Fort Sheridan, Pittsburgh, and Annapolis Tests.. 97
  Report of Sub-Committee V on Total Immersion Tests.................... 99
  Report of Sub-Committee VI on Specifications for Metallic-Coated Products 101
    Appendix. Proposed Requirements for Zinc-Coated or Galvanized Wire. 102
  Report of Sub-Committee VII on Accelerated Tests...................... 103
  Report of Sub-Committee VIII on Tests of Metallic-Coated Products..... 104
Report of Committee A-7 on Malleable Castings............................ 118
  Discussion.. 119
Report of Committee A-9 on Ferro-Alloys.................................. 121
  Discussion.. 123

Non-Ferrous Metals

Report of Committee B-1 on Copper Wire................................... 126
Report of Committee B-2 on Non-Ferrous Metals and Alloys................. 129
  Appendix. Review of the Antimony Situation—H. K. Masters.............. 143
  Addendum to Report of Committee B-2.................................. 149
Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys.... 150

Cement, Lime, Gypsum, and Clay Products

Report of Committee C-1 on Cement.. 159
  Appendix. Manual of Cement Testing.................................... 161
  Discussion.. 178

PAGE
Report of Committee C-3 on Brick.......... 184
Report of Committee C-7 on Lime.......... 186
Report of Committee C-8 on Refractories.......... 190
Appendix. Report on Industrial Survey.......... 192
Report of Committee C-9 on Concrete and Concrete Aggregates.......... 199
Appendix I. Digest of Literature on Methods of Making Flexure and Tension Tests of Concrete.......... 205
Appendix II. Concrete Aggregate Material Investigation Report.......... 209
Report of Committee C-10 on Hollow Masonry Building Units.......... 210
Report of Committee C-11 on Gypsum.......... 211
Appendix. Proposed Revisions of the Standard Methods of Testing Gypsum and Gypsum Products.......... 216
Discussion.......... 218

Miscellaneous Materials: Preservative Coatings, etc.

Report of Committee D-1 on Preservative Coatings for Structural Materials... 220
Report of Sub-Committee III on Testing of Paint Vehicles.......... 227
Report of Sub-Committee VI on Definitions of Terms Used in Paint Specifications.......... 232
Report of Sub Committee IX on Varnish.......... 233
Report of Sub-Committee XIV on Preparation of Iron and Steel Surfaces for Painting.......... 238
Report of Sub-Committee XVIII on Physical Properties of Paint Materials 241
Report of Sub-Committee XXII on Method of Application of Paint by Spraying.......... 243
Report of Sub-Committee XXIII on Anti-Fouling Paints.......... 244
Report of Committee D-2 on Petroleum Products and Lubricants.......... 248
Report of Sub-Committee III on Paraffin Wax.......... 260
Appendix. Proposed Method for Determination of Oil in Wax.......... 263
Report of Sub-Committee IV on Grease.......... 266
Report of Sub-Committee VII on Sulfur Determination and Differentiation 269
Report of Sub-Committee XI on Emulsification.......... 272
Report of Sub-Committee XIII on Neutralization Number and Saponification.......... 280
Appendix. Proposed Method of Test for Neutralization Number of Petroleum Oil, Titration by Electrometric Method.......... 282
Report of Sub-Committee XV on Sampling and Gaging.......... 286
Report of Sub-Committee XX on Nomenclature.......... 287
Report of Sub-Committee XXI on Crude Petroleum.......... 290
Report of Sub-Committee XXIV on Calorific Value of Fuel Oil.......... 292
Report of Committee D-4 on Road and Paving Materials.......... 293
Appendix. Report of Sectional Committee on Penetration.......... 301
Report of Committee D-5 on Coal and Coke.......... 307
Report of Committee D-7 on Timber.......... 310
Report of Sub-Committee I on Specifications for Timber.......... 312
Appendix. Working Stresses for Douglas Fir and Southern Pine.......... 335
Report of Sub-Committee VI on Timber Preservatives.......... 340
Report of Committee D-8 on Bituminous Waterproofing and Roofing Materials 342

PAGE
Report of Committee D-9 on Electrical Insulating Materials.................. 348
  Appendix. Life Test for Transformer Oils—E. A. Snyder and D. C. Cox. 363
  Discussion.. 369
Report of Committee D-11 on Rubber Products.......................... 371
Report of Committee D-13 on Textile Materials.......................... 376
Report of Committee D-14 on Screen Wire Cloth.......................... 384
Report of Committee D-15 on Thermometers.............................. 391
  Proposed Specifications for A.S.T.M. Low-Cloud and Pour Test Thermometers −60 to +20° C., −70 to +70° F.......................... 393
Report of Committee D-16 on Slate.................................. 395

Miscellaneous Subjects

Report of Committee E-1 on Methods of Testing.......................... 398
  Report of Sub-Committee on Determination of Water.................. 416
  Appendix I. Proposed Methods of Flexure Testing of Metallic Materials. 424
  Appendix II. Proposed Methods for Torsion Tests to Determine the Mechanical Properties of Metallic Materials Under Shearing Stress........ 430
  Discussion.. 437
Report of Committee E-4 on Metallography.............................. 440
  Report of Sub-Committee V on Micro-Hardness.......................... 442
  Report of Sub-Committee VI on X-ray Metallography.................. 444
  Discussion.. 470
Report of Committee E-6 on Papers and Publications.................... 486
Report of Committee E-8 on Nomenclature and Definitions................ 488
Report of Committee E-9 on Correlation of Research...................... 502

A.S.T.M. TENTATIVE STANDARDS

Submitted or Revised at the 1925 Annual Meeting

A. FERROUS METALS

Tentative Specifications for:

A 21 − 25 T. Carbon-Steel Car and Tender Axles.................... 509
A 94 − 25 T. Structural Silicon Steel.............................. 514
A 95 − 25 T. Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service.......................... 519
A 96 − 25 T. Alloy-Steel Bolting Material for High-Temperature Service 523
A 97 − 25 T. Tungsten Powder.................................... 528
A 98 − 25 T. Spiegeleisen.. 529
A 99 − 25 T. Ferro-Manganese.................................... 530
A 100 − 25 T. Ferro-Silicon...................................... 531
A 101 − 25 T. Ferro-Chromium.................................... 532
A 102 − 25 T. Ferro-Vanadium.................................... 533
A 86 − 25 T. Hollow Staybolt Iron.................................. 534

Tentative Methods of:

A 103 − 25 T. Sampling Ferro-Alloys.............................. 537
A 104 − 25 T. Chemical Analysis of Ferro-Alloys.................... 542

Tentative Recommended Practice for:

A 37 − 25 T. Carburizing and Heat Treatment of Carburized Objects.. 568

B. NON-FERROUS METALS

Tentative Specifications for:

PAGE
B 26–25 T. Aluminum Base Alloy Sand Castings 574
B 9–25 T. Bronze Trolley Wire 578
B 57–25 T. Muntz Metal Condenser Tube Plates 583
B 24–25 T. Aluminum Ingots for Remelting 585
B 50–25 T. Non-Ferrous Insect Screen Cloth 587

Tentative Methods of:

B 40–25 T. Chemical Analysis of Aluminum and Light Aluminum Alloys 590

C. CEMENT, LIME, GYPSUM, AND CLAY PRODUCTS

Tentative Specifications for:

C 46–25 T. Quicklime for Use in the Manufacture of Sulfite Pulp 612
C 53–25 T. Quicklime for Use in Water Treatment 614
C 54–25 T. Hydrated Lime for Use in Water Treatment 616

Tentative Methods of:

C 25–25 T. Chemical Analysis of Limestone, Quicklime and Hydrated Lime 618
C 42–25 T. Securing Specimens of Hardened Concrete from the Structure 631

Tentative Definitions of:

C 11–25 T. Terms Relating to the Gypsum Industry 633
C 58–25 T. The Term Sand 635

D. MISCELLANEOUS MATERIALS

Preservative Coatings

Tentative Specifications for:

D 12–25 T. Raw Tung Oil 636
D 207–25 T. Dry Bleached Shellac 644
D 84–25 T. Iron Oxide and Iron Hydroxide 646
D 208–25 T. Lithopone 648
D 209–25 T. Lampblack 650
D 210–25 T. Bone Black 652
D 211–25 T. Chrome Yellow 654
D 212–25 T. Pure Chrome Green 656
D 213–25 T. Reduced Chrome Green 658

Tentative Methods of:

D 214–25 T. Testing Shellac Varnish 660
D 215–25 T. Routine Analysis of White Linseed Oil Paints 664

Petroleum Products and Lubricants

Tentative Methods of:

D 86–25 T. Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products 685
D 216–25 T. Test for Distillation of Natural Gas Gasoline 693

Tentative Methods of:

PAGE
D 217 – 25 T. Test for Penetration of Greases........ 701
D 88 – 25 T. Test for Viscosity of Petroleum Products and Lubricants. 707
D 218 – 25 T. Test for Color of Petrolatum by Means of the Union Colorimeter........ 713
D 219 – 25 T. Test for Burning Quality of Long-Time Burning Oil for Railway Use........ 718
D 188 – 25 T. Test for Neutralization Number of Petroleum Products and Lubricants........ 722
D 97 – 25 T. Test for Cloud and Pour Points of Petroleum Products... 725
D 158 – 25 T. Testing Gas Oils (Gravity, Distillation, Sulfur, Carbon Residue, Pour Point, Viscosity, Water)........ 732

Road Materials

Tentative Specifications for:

D 106 – 25 T. High-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product)........ 738
D 110 – 25 T. High-Carbon Tar Cement........ 740

Tentative Methods of:

D 137 – 25 T. Mechanical Analysis of Subgrade Soils........ 742
D 220 – 25 T. Test for the Determination of Moisture Equivalent of Subgrade Soils in the Field........ 745
D 138 – 25 T. Test for Consistency of Portland-Cement Concrete........ 746
D 139 – 25 T. Float Test for Bituminous Materials........ 748

Coal

Tentative Definitions of:

D 142 – 25 T. Terms Relating to Coal........ 752

Slate

Tentative Methods of:

D 221 – 25 T. Test for Water Absorption of Slate........ 754
D 222 – 25 T. Flexure Testing of Slate (Determination of Modulus of Rupture and Modulus of Elasticity)........ 756

Waterproofing

Tentative Specifications for:

D 223 – 25 T. Acid-Resisting Asphalt Mastic........ 759
D 224 – 25 T. Smooth-Surfaced Asphalt Roll-Roofing........ 761
D 225 – 25 T. Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles........ 765
D 226 – 25 T. Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs........ 770
D 227 – 25 T. Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs........ 773

Tentative Methods of:

D 146 – 25 T. Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing........ 776

Tentative Methods of: PAGE
D 147 – 25 T. Testing Bituminous Mastics, Grouts and Like Mixtures.. 786
D 228 – 25 T. Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles........ 792

Insulating Materials

Tentative Methods of:
D 115 – 25 T. Testing Insulating Varnishes........ 797
D 175 – 25 T. Testing Electrical Insulating Materials for Voltage Effects at Radio Frequencies........ 804
D 176 – 25 T. Testing Cable Splicing and Pothead Compounds........ 810
D 202 – 25 T. Testing Untreated Insulating Paper........ 815
D 229 – 25 T. Testing Laminated Sheet Insulating Materials........ 825

Textile Materials

Tentative Specifications for:
D 76 – 25 T. Textile Testing Machines........ 829
D 180 – 25 T. Tolerances and Test Methods for Cotton Yarns, Single and Plied........ 831
D 204 – 25 T. Tolerances and Test Methods for Cotton Sewing Threads. 837
D 230 – 25 T. Tolerances for Numbered Cotton Duck........ 840
D 231 – 25 T. Tolerances and Test Methods for Knit Goods........ 842

Tentative Methods of:
D 232 – 25 T. Testing Grease Wool and Allied Fibers for Scoured Content 846

E. MISCELLANEOUS SUBJECTS

Tentative Specifications for:
E 11 – 25 T. Sieves for Testing Purposes........ 850

Tentative Methods of:
E 8 – 25 T. Tension Testing of Metallic Materials........ 854
E 9 – 25 T. Compression Testing of Metallic Materials........ 867
E 10 – 25 T. Brinell Hardness Testing of Metallic Materials........ 872

Tentative Definitions of:
E 6 – 25 T. Terms Relating to Methods of Testing........ 879
E 12 – 25 T. Terms Relating to Specific Gravity........ 887
E 13 – 25 T. The Term Screen (Sieve)........ 889

Tentative Recommended Practice for:
E 14 – 25 T. Thermal Analysis of Steel........ 890

TENTATIVE REVISIONS OF A.S.T.M. STANDARDS

A. FERROUS METALS

Standard Specifications for:
A 18 – 21. Carbon-Steel and Alloy-Steel Forgings........ 895
A 19 – 21. Quenched-and-Tempered Carbon-Steel Axles, Shafts and Other Forgings for Locomotives and Cars........ 895

Standard Specifications for:

PAGE
A 20 – 21. Carbon-Steel Forgings for Locomotives 895
A 63 – 21. Quenched-and-Tempered Alloy-Steel Axles, Shafts and Other Forgings for Locomotives and Cars 895
A 21 – 18. Carbon Steel Car and Tender Axles 896
A 53 – 24. Welded and Seamless Steel Pipe 896
A 83 – 24. Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes 896
A 72 – 24. Welded Wrought-Iron Pipe 897
A 84 – 24. Staybolt, Engine-Bolt and Extra-Refined Wrought-Iron Bars. 897
A 47 – 24. Malleable Castings 897

Recommended Practice for:

A 37 – 14. Heat Treatment of Case-Hardened Carbon-Steel Objects 898

B. NON-FERROUS METALS

Standard Specifications for:

B 4 – 13. Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars 898
B 5 – 13. Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars 901
B 26 – 21. Light Aluminum Casting Alloys 902
B 9 – 16. High-Strength Bronze Trolley Wire, Round and Grooved, 40 and 65-per-cent Conductivity 902
B 44 – 24. Seamless Admirality Condenser Tubes and Ferrule Stock 902
B 36 – 21. High Sheet Brass 903
B 21 – 19. Naval Brass Rods for Structural Purposes 903

C. CEMENT, LIME, GYPSUM, AND CLAY PRODUCTS

Standard Specifications for:

C 9 – 21. Specifications and Tests for Portland Cement 904
C 14 – 24. Cement-Concrete Sewer Pipe 904
C 19 – 18. Fire Tests of Materials and Construction 904
C 7 – 15. Paving Brick 907
C 6 – 24. Hydrated Lime for Structural Purposes 907

Standard Methods of:

C 26 – 23. Testing Gypsum and Gypsum Products 908

D. MISCELLANEOUS MATERIALS

Standard Specifications for:

D 13 – 24. Turpentine 909
D 65 – 23. Broken Slag for Waterbound Base and Wearing Course 910
D 66 – 23. Shovel-Run or Crusher-Run Broken Slag for Waterbound Base 910

Standard Methods of:

D 20 – 18. Test for Distillation of Bituminous Materials Suitable for Road Treatment 911
D 37 – 24. Laboratory Sampling and Analysis of Coke 911

Standard Methods of:

PAGE
D 22 – 24. Laboratory Sampling and Analysis of Coal.................. 912
D 38 – 24. Sampling and Analysis of Creosote Oil.................... 912
D 48 – 24. Testing Molded Insulating Materials...................... 912

Standard Definitions of:

D 16 – 24. Terms Relating to Paint Specifications.................... 917

Officers and Members of Executive Committee.......................... 918
Subject Index.. 919
Author Index... 939
List of Standards.. 945
List of Tentative Standards.. 955

INSERT PLATE

I. Total Immersion Tests.. 100

SUMMARY OF THE PROCEEDINGS OF THE TWENTY-EIGHTH ANNUAL MEETING

Atlantic City, N. J., June 23–26, 1925

The Twenty-eighth Annual Meeting of the American Society for Testing Materials was held at the Chalfonte-Haddon Hall, Atlantic City, N. J., June 23–26, 1925. The following is an analysis of the attendance at the meeting: Members in attendance or represented, 775; guests, 93; total, 868; ladies, 250. The corresponding statistics for the Twenty-seventh Annual Meeting are: Members in attendance or represented, 719; guests, 143; total, 862; ladies, 206.

First Session—Tuesday, June 23, 8 p. m.

On Wrought Iron, Cast Iron and Corrosion

President F. M. Farmer in the chair.

The minutes of the Twenty-seventh Annual Meeting were approved as printed.

The report of Committee A-2 on Wrought Iron was presented by the chairman, H. J. Force. On recommendation of the committee, revisions of the Standard Specifications for Welded Wrought-Iron Pipe (A 72 – 24), the Standard Specifications for Staybolt, Engine-Bolt and Extra-Refined Wrought-Iron Bars (A 84 – 24), and of the Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 24) were accepted for publication as tentative.

Revisions recommended by the committee in the Tentative Specifications for Hollow Staybolt Iron (A 86 – 21 T) were accepted, the specifications being continued as tentative.

The report of the committee was, on motion, adopted, including the Methods of Sampling and Chemical Analysis of Wrought Iron, presented as information.

The report of Committee A-3 on Cast Iron was presented by the chairman, Richard Moldenke. The report contained a statement that the committee had reached the decision to change the dimensions of the arbitration test bar. It was not intended, however, to recommend any revisions at the present time of the standard specifications in which the requirements for the arbitration test bar appear. These revisions would be developed by the committee during the coming year. This subject received some discussion on the floor of the meeting. On motion, the report was adopted.

The report of Committee A-7 on Malleable Castings, in the absence of the chairman, W. P. Putman, was presented by the secretary, Enrique Touceda. Proposed revisions of the Standard Specifications for Malleable Castings (A 47 – 24), after some discussion, were accepted for publication as tentative as recommended by the committee. On motion, the report was adopted.

The report of Committee A-5 on Corrosion of Iron and Steel was presented by the chairman, J. H. Gibboney. This report consisted of a report of progress on the inspection of the Fort Sheridan, Pittsburgh and Annapolis atmospheric exposure tests and on total immersion tests and submitted as information Proposed Requirements for Zinc-Coated and Galvanized Wire. In addition, a sub-committee under the chairmanship of F. A. Hull presented an outline of contemplated atmospheric exposure tests of metallic-coated products. The chairman called on Mr. Hull to present the report of his subcommittee and on H. S. Rawdon, chairman of the Sub-Committee on Accelerated Tests, to present the report of his sub-committee on accelerated tests to be carried out in conjunction with the long-time exposure tests. The report of the committee was, on motion, adopted.

A paper entitled "Accelerated Corrosion Tests on Bare Overhead Electrical Conductors" by Frank F. Fowle was presented by the author and discussed.

The report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, E. C. Lathrop, chairman, was presented by the secretary, Sam Tour. The report had not been preprinted and was accordingly presented from manuscript. On motion, the report of the committee was adopted.

A paper entitled "The Evaluation of Corrosion Tests" by E. Blough was presented by the author and discussed.

The meeting then adjourned till the following morning.

SECOND SESSION—WEDNESDAY, JUNE 23, 9.30 A. M.

On Non-Ferrous Metals and Metallography

(Held Simultaneously with the Third Session)

Vice-President J. H. Gibboney in the chair.

The report of Committee B-1 on Copper Wire was presented by the chairman, J. A. Capp. The Tentative Specifications for Round and Grooved Hard-Drawn Copper Trolley Wire (B 47 – 23 T), revised as appended to the report of the committee, were, on recommendation of the committee, referred to letter ballot of the Society for adoption as standard. These specifications represented final and complete agreement between this Society and the American Electric Railway Engineering Association on specifications for hard-drawn copper trolley wire.

The committee had been cooperating with the A.E.R.E.A. on the preparation of specifications for bronze trolley wire and submitted proposed Tentative Specifications for Bronze Trolley Wire which, with the exception of one requirement, were in agreement with the specifications of the A.E.R.E.A. On recommendation of the committee these specifications were accepted for publication as tentative to supersede, when adopted, the present Standard Specifications for High-Strength Bronze Trolley Wire, Round and Grooved: 40 and 65-per-cent Conductivity (B 9 – 16). The report of the committee was, on motion, adopted.

A paper entitled "Softening of Hard-Rolled Electrolytic Copper," by Norman B. Pilling and George P. Halliwell, was presented by Mr. Pilling and discussed.

The report of Committee B-2 on Non-Ferrous Metals and Alloys, in the absence of the chairman, William Campbell, was presented by the vice-chairman, W. R. Webster. The revisions of the Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars (B 4 – 13), Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 – 13), Standard Specifications for High Sheet Brass (B 36 – 21) and Standard Specifications for Naval Brass Rods for Structural Purposes (B 21 – 19) were, on recommendation of the committee, accepted for publication as tentative. The revisions of the Standard Specifications for Seamless Admiralty Condenser Tubes and Ferrule Stock (B 44 – 24) were accepted for publication as tentative subject to letter ballot of the committee, since through an oversight this revision had not been sent out to letter ballot in advance of the meeting.[1]

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[1] Results of this letter ballot have since been reported as follows: Affirmative, 34; negative, 0; not voting, 10.—ED.

A footnote covering the use of virgin metals in the Tentative Specifications for Manganese-Bronze Ingots for Sand Castings (B 7 - 24 T) was added to the specifications in accordance with the recommendation appearing in the Addendum to Report of Committee B-2.

The revisions recommended by the committee in the following tentative standards were accepted, the tentative standards as revised being continued as tentative: the Tentative Specifications for Light Aluminum Casting Alloys (B 26 - 24 T), Tentative Specifications for Aluminum Ingots for Remelting and for Rolling (B 24 - 22 T), and the Tentative Methods of Chemical Analysis of Light Aluminum Alloys (B 40 - 23 T).

The proposed Tentative Specifications for Muntz Metal Condenser Tubes Plates, on recommendation of the committee, were accepted for publication as tentative.

The Tentative Specifications for Seamless 70-30 Brass Condenser Tubes and Ferrule Stock (B 55 - 24 T) and the Tentative Specifications for Seamless Muntz Metal Condenser Tubes and Ferrule Stock (B 56 - 24 T) were referred to letter ballot of the Society for adoption as standard as recommended by the committee. On motion, the report of the committee was adopted, including a paper by H. K. Masters entitled "A Review of the Antimony Situation" submitted by the committee as information.

A paper entitled "A Note on the Microstructure of Aluminum-Iron Alloys of High Purity," by E. H. Dix, Jr., was presented by the author and discussed.

The report of Committee D-14 on Screen Wire Cloth was presented by the chairman, R. W. Woodward. The revision recommended by the committee in the Tentative Specifications for Non-Ferrous Insect Screen Cloth (B 50 - 23 T) was accepted, the specifications being continued as tentative. The report of the committee was, on motion, adopted.

The report of Committee E-4 on Metallography, in the absence of the chairman, H. C. Boynton, was presented by the secretary, G. F. Comstock. The proposed Recommended Practice for Thermal Analysis of Steel was accepted for publication as tentative as recommended by the committee. The chairman then called on Zay Jeffries to present the report of the Sub-Committee on X-ray Metallography, which report was presented from manuscript and discussed. On motion, the report of the committee was adopted.

The meeting then adjourned till 8 P. M.

THIRD SESSION, WEDNESDAY, JUNE 24, 9.30 A. M.

On Ceramics, Coal, Timber, Rubber and Slate

(Held Simultaneously with the Second Session)

Vice-President W. H. Fulweiler in the chair.

The report of Committee C-3 on Brick was presented by the chairman, T. R. Lawson. The proposed revision of the Standard Specifications for Paving Brick (C 7 – 15) consisting of the elimination of one more size, leaving four recognized sizes and types of brick in accordance with the recommendations of the Conference on Elimination of Unnecessary Sizes and Types of Paving Brick, organized by the U. S. Department of Commerce, was accepted for publication as tentative. By vote of the meeting, the action of a similar Conference on standard sizes for sand-lime brick, in fixing the recommended size of brick, was approved, the size being the same as that now called for in the Society's Standard Specifications for Building Brick (C 21 – 20). The report of the committee was, on motion, adopted.

A paper entitled "Brick," by D. Knickerbacker Boyd, was presented by the author and discussed.

The report of Committee C-10 on Hollow Masonry Building Units was presented by the chairman, S. H. Ingberg, and discussed. On motion, the report was adopted.

The report of Committee D-16 on Slate was presented, in the absence of the chairman, D. W. Kessler, by the secretary, D. K. Boyd. The proposed Tentative Method of Test for Water Absorption of Slate and the proposed Tentative Methods of Flexure Testing of Slate were accepted for publication as tentative as recommended by the committee, with the addition of the following footnote to the Methods of Flexure Testing:

> "The shapes of specimens recommended for modulus of rupture and elasticity tests were determined partly by consideration of the proportions best adapted to the respective tests and partly on account of the convenience of preparing same from stock material. The small sizes recommended were first for economy of material and second, to permit the use of lighter testing equipment. Where only the large machines are available it is desirable, in order to obtain consistent results, to use larger specimens. In such cases approximately the same proportionate dimensions should be adhered to for the respective tests."

The report of Committee C-8 on Refractories (W. H. Fulweiler, chairman), was presented by the secretary, F. A. Harvey. On recommendation of the committee, the withdrawal of the Tentative Method of Test for Slagging Action of Refractory Materials (C 17 – 19 T) was approved. On motion, the report of the committee was adopted, including the report on Industrial Survey, covering the Malleable Iron Industry and the By-Product Coke Oven Industry, appended to the committee's report.

The report of Committee D-5 on Coal and Coke was presented by the chairman, A. C. Fieldner. The revisions recommended by the committee in the Standard Methods of Laboratory Sampling and Analysis of Coke (D 37 – 24) and in the Standard Methods of Laboratory Sampling and Analysis of Coal (D 22 – 24) were approved for publication as tentative. The revisions in the Tentative Definitions of Terms Relating to Coal (D 142 – 24 T) recommended by the committee, were accepted, the definitions being continued as tentative. On motion, the report was adopted.

The report of Committee D-7 on Timber was presented, in the absence of the chairman, Hermann von Schrenk, by C. E. Paul. This presentation included the report of the Sub-Committee on Specifications for Timber which had not been preprinted in advance of the meeting. The committee had, however, indicated its intention of presenting this report and had asked leave to have the report presented verbally, the final form to be developed subsequent to the annual meeting, this report to cover the revision in the proposed specifications submitted as information last year to bring these specifications in line with the agreements reached at a conference on lumber standards called by the Secretary of Commerce. On motion, the report of the committee, including the revised specifications for timber to be published as information, was adopted.

The report of Committee D-11 on Rubber Products was presented by the chairman, F. M. Farmer. The report as preprinted contained a recommendation that a test requirement covering adhesiveness be added to the Tentative Specifications for Rubber Insulating Tape (D 119 – 22 T). This recommendation of the committee was withdrawn. The report as amended was, on motion, adopted.

The meeting adjourned till 8 P. M.

FOURTH SESSION—WEDNESDAY, JUNE 24, 8 P. M.

Presidential Address and Reports of Administrative Committees

Past-President G. H. Clamer in the chair.

The chairman introduced President F. M. Farmer, who presented the annual presidential address, choosing for the topic of his address "The Promotion of Knowledge of Engineering Materials."

The secretary presented the annual report of the Executive Committee which discussed membership activities, the work of standing committees, publications, finances and administrative matters relating to committee activities and relations with other socities.

In the Executive Committee report are the names of the members who have died during the past year. One of those who had died recently was the first chairman of the American Section of the International Association for Testing Materials, Mansfield Merriman. The following minute was adopted by a standing and silent vote:

MANSFIELD MERRIMAN

1848–1925

The American Society for Testing Materials in annual meeting assembled desires to record its profound sorrow in the death, on June 7, 1925, of its honored Past-President and Honorary Member, Mansfield Merriman.

His life was in large part devoted to the teaching of engineering and to the writing of engineering textbooks, in which he attained great eminence that brought many honors and recognition to him. He was a leader in the development of engineering education in this country. He was highly renowned as an investigator and teacher, and he greatly enriched the engineering literature of his day.

Professor Merriman was a leader in our field of testing the materials of engineering. He was interested from its very inception in the activities of the Society and contributed much to its development and progress. He was one of the influential American members of the International Association for Testing Materials and was one of a group that brought about the organization of the American Section of the International Association for Testing Materials in 1898. As the first chairman of that Section from 1898 to 1900 and as a member of its Executive Committee from 1900 to 1902, his leadership in those early days of doubt and uncertainty was instrumental in laying the foundation upon which our Society was organized as a separate body in 1902. As President in 1915, he was again called to the highest office in the Society, and in 1919 he was made an Honorary Member of the Society. In every way the Society has benefited and been honored by his membership.

His death brings a deep sense of personal loss to all who were privileged to know him. The Society mourns the loss of so distinguished and honored a member and extends its heartfelt sympathy to his family.

It was reported that the Executive Committee had given consideration during the year to a suggestion received from Committee E-9 on Correlation of Research that (1) an annual medal be established to be awarded for a paper of outstanding merit, constituting an original contribution on research in materials, and (2) the establishment of an annual lecture to be given at the annual meeting by leaders in the field of engineering materials. The establishment of a medal to be known as the Charles B. Dudley Medal, commemorating the name of the first president of the Society, the late Charles B. Dudley, President from 1902 until 1909, and a lecture known as the Edgar Marburg Lecture, commemorating the name of Edgar Marburg, secretary of the Society from 1902 until his death in 1918, was accordingly proposed. This proposal met with the hearty approval of the members assembled. The motion to establish a medal and lecture was made by W. H. Fulweiler, seconded by George S. Webster, and further supported by A. N. Talbot. After the adoption of the motion the announcement was made that the Executive Committee wished to head the subscription list in raising the necessary funds and had contributed $390.

The Executive Committee further called special attention to the organization of a committee on Promotion of Usefulness of the A.S.T.M. This special committee is under the chairmanship of J. H. Gibboney and consists of the following:

Chandler, W. L.
Farmer, F. M. (*ex-officio*)
Gibboney, J. H.
Kinney, W. M.
Lynch, T. D.
Marston, Anson
Rys, C. F. W.
Warwick, C. L. (*ex-officio*)
Wiggin, T. H.

On motion, the report of the Executive Committee was adopted.

The report of Committee E-6 on Papers and Publications was presented by the chairman, C. L. Warwick. On motion, the report of the committee was adopted.

The report of the committee of tellers consisting of H. J. Force and H. C. Porter, appointed to canvass the letter ballot of election of officers, was received. The tellers reported that 779 legal ballots had been cast, the vote being as follows:

For President, to serve for one year: W. H. Fulweiler, 776 votes.
For Vice-President, to serve for two years: H. F. Moore, 778 votes.
For Members of Executive Committee, to serve for two years:

Louis Anderson, Jr., 775 votes.
E. F. Kenney, 775 votes.
T. D. Lynch, 777 votes.
K. G. Mackenzie, 775 votes.

In accordance with the ballot, the Chair declared the officers listed above, elected.

The chairman then requested the Past-Presidents George S. Webster and A. N. Talbot to escort the President-elect to the chair.

The President-elect, W. H. Fulweiler, expressed his appreciation of the honor of his election to the Presidency and appealed to the members for their cooperation in carrying on the duties connected with the office.

The Vice-President elect, H. F. Moore, was then escorted to the chair by Past-Presidents George S. Webster and A. N. Talbot. After acknowledging the honor of his election he expressed the hope that he might have the opportunity to participate in carrying out some of the plans under discussion for advancing the work of the Society.

The meeting then adjourned till the following morning and was followed by the annual dance and smoker.

FIFTH SESSION—THURSDAY, JUNE 25, 9.30 A. M.

On Steel and Fatigue of Metals

(Held Simultaneously with the Sixth Session)

Past-President C. D. Young in the chair.

The report of Committee A-1 on Steel was presented by the chairman, F. M. Waring. The proposed revisions in the following standard specifications were accepted for publication as tentative:

Standard Specifications for:

Carbon-Steel and Alloy-Steel Forgings (A 18 - 21);
Quenched-and-Tempered Carbon-Steel Axles, Shafts and Other Forgings for Locomotives and Cars (A 19 - 21);
Carbon-Steel Forgings for Locomotives (A 20 - 21);
Quenched-and-Tempered Alloy-Steel Axles, Shafts and Other Forgings for Locomotives and Cars (A 63 - 21);
Welded and Seamless Steel Pipe (A 53 - 24);
Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 - 24).

The rewriting of the Standard Specifications for Carbon-Steel Car and Tender Axles (A 21 - 18) was accepted for publication as a tentative standard as appended to the report of the committee, to replace, when adopted, the standard specifications of the same title. The proposed new Specifications for Structral Silicon Steel, for Carbon-Steel Castings for Valves, Flanges and Fittings for High-Temperature Service and for Alloy-Steel Bolting Material for High-Temperature Service were accepted for publication as tentative as recommended by the committee. On motion, the report of the committee was adopted.

The report of Committee A-4 on Heat Treatment of Iron and

Steel, in the absence of the chairman, H. M. Boylston, was presented by A. H. Jameson. The new Recommended Practice for Carburizing and Heat Treatment of Carburized Objects, proposed by the committee, was accepted for publication as tentative, to replace, when adopted, the present Recommended Practice for Heat Treatment of Case-Hardened Carbon-Steel Objects (A 37 – 14). The report of the committee was, on motion, adopted.

The report of Committee A-9 on Ferro-Alloys was presented by the chairman, F. C. Langenberg. The chairman then called on N. B. Hoffman to present the several proposed specifications recommended by the committee. These specifications include the following:

Tentative Specifications for Tungsten Powder;
Tentative Specifications for Spiegeleisen;
Tentative Specifications for Ferro-Silicon;
Tentative Specifications for Ferro-Chromium;
Tentative Specifications for Ferro-Manganese;
Tentative Specifications for Ferro-Vanadium.

In submitting these specifications several modifications were recommended, namely, the addition under "Basis of Purchase" in the Specifications for Ferro-Vanadium of the requirement "The material shall be packed in sound containers sufficiently strong to prevent loss in transportation," and the change in the manganese requirement for Grade A in the Specifications for Spiegeleisen to read "19–21 per cent" instead of "19–22 per cent." In the Specifications for Spiegeleisen as preprinted, a maximum requirement for silicon of 1.5 per cent was given in the case of Grade A, and of 2.0 per cent for Grade B material. It was recommended that this requirement be replaced by the requirement that the maximum silicon be as specified. The phrase "and each shipment shall be uniform as to the grade specified," was added to Section 2 in "Basis of Purchase." On motion, the specifications as modified were accepted, after some discussion, for publication as tentative. The proposed Methods of Sampling Ferro-Alloys were presented by J. A. Holladay and, on motion, were accepted for publication as tentative.

The proposed Methods of Chemical Analysis of Ferro-Alloys were presented by G. E. F. Lundell. The Methods as preprinted contained methods for the determination of tin, bismuth, antimony and copper and for the determination of arsenic. It was recommended by the committee that the sections covering these methods be withdrawn. With some few additional minor corrections, the proposed Tentative Methods of Chemical Analysis of Ferro-Alloys were, on motion, accepted for publication as tentative. The report, on motion, was adopted.

A paper entitled "Fatigue of Metals by Direct Stress" by Paul L. Irwin, and a paper entitled "Some Fatigue Tests on Non-Ferrous Metals" by R. R. Moore, were presented by the authors and discussed jointly.

A paper entitled "Tensile Properties of Metals at High Temperature Showing Effect of Stress and Time," by T. D. Lynch, P. J. McVetty and N. L. Mochel, was presented by Mr. McVetty, and a paper by T. McL. Jasper entitled "Typical Static and Fatigue Tests on Steel at Elevated Temperatures," was presented by the author, and the two papers were discussed jointly.

The meeting adjourned till 8 P. M.

SIXTH SESSION—THURSDAY, JUNE 25, 9.30 A. M.

On Road Materials and Waterproofing and Roofing Materials

(*Held Simultaneously with the Fifth Session*)

T. G. Delbridge in the chair.

The report of Committee D-4 on Road and Paving Materials was presented by the chairman, H. S. Mattimore. The proposed revision of the Standard Method of Test for Penetration of Bituminous Materials (D 5 – 21) was unanimously accepted by the meeting for reference to letter ballot of the Society for adoption as standard as recommended by the committee. Since it had been recommended to advance this revision to standard immediately without its being published as tentative, the committee had asked for the necessary nine-tenths vote required by the regulations of the Society.

A similar nine-tenths vote was asked on the proposed revision of the Standard Methods of Test for Apparent Specific Gravity of Sand, Stone and Slag Screenings, and Other Fine Non-Bituminous Highway Materials (D 55 – 24). Some minor modifications in the wording of Section 3 (*a*) of the Standard Methods, all of an editorial nature, were made from the wording given in the report as preprinted. On motion, the revision of the Standard Methods D 55 – 24 was unanimously accepted by the meeting for reference to letter ballot of the Society for adoption as standard as recommended by the committee.

The committee, in the report as preprinted, had recommended a revision of the Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (D 20 – 18), the revision to be adopted immediately. This recommendation was changed by the committee for the acceptance of the revision for publication as tentative, rather than for its adoption. On motion, this recommendation was approved and the revision accepted for publication as tentative.

The proposed revisions of the following Tentative Specifications and Methods of Test were accepted for publication as tentative as recommended by the committee:

Tentative Specifications for:

High-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product) (D 106 – 23 T);
High-Carbon Tar Cement (D 110 – 23 T).

Tentative Methods of:

Float Test for Bituminous Materials (D 139 – 24 T);
Mechanical Analysis of Subgrade Soils (D 137 – 24 T);
Test for Consistency of Portland Cement Concrete for Pavements or for Pavement Base (D 138 – 22 T).

The proposed new Method of Test for Determination of Moisture Equivalent of Subgrade Soils in the Field was accepted for publication as tentative as recommended by the committee.

The Tentative Methods of Sampling Bituminous Materials (D 140 – 23 T) were, on recommendation of the committee, referred to letter ballot of the Society for adoption as standard. On motion, the report of the committee was adopted.

The following papers were then presented:

"A Practical Method for Determining the Relative Stability of Fine-Aggregrate Asphalt Paving Mixtures," by Prévost Hubbard and F. C. Field—presented by Mr. Hubbard.
"A Stability Test for Bituminous Paving Mixtures," by W. J. Emmons and B. A. Anderton—presented by Mr. Anderton.
"A Deformation Test for Asphaltic Mixtures," by Henry M. Milburn—presented by the author.

These three papers, all dealing with the subject of stability of bituminous mixtures, were discussed jointly.

A paper by F. L. Sperry on "Quality of Oil for Surface Oiling of Earth Roads and Streets," was read by title.

The report of Committee D-8 on Bituminuous Waterproofing and Roofing Materials in the absence of the chairman, S. T. Wagner, was presented by the vice-chairman, C. N. Forrest. The proposed revisions of the Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (D 147 – 24 T), and of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 146 – 23 T) were accepted as recommended by the committee, the methods being continued as tentative. The following proposed new tentative standards were accepted as recommended by the committee, for publication as tentative:

Tentative Specifications for:

Acid-Resisting Asphaltic Mastic;
Smooth-Surfaced Asphalt Roll Roofing;
Slate-Surfaced Asphalt Roll Roofing and Slate-Surfaced Asphalt Shingles;
Asphalt-Saturated Roofing Felt for Use in Waterproofing and Constructing Built-Up Roofs;
Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs.

Tentative Methods of:

Testing Smooth-Surfaced Asphalt Roll Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles.

On recommendation of the committee the following tentative standards were referred to letter ballot of the Society for adoption as standard:

Tentative Specifications for:

Asphalt Mastic for Use in Waterproofing (D 169 – 24 T);
Bituminous Grout for Use in Waterproofing Above Ground Level (D 170 – 24 T);
Bituminous Grout for Use in Waterproofing Below Ground Level (D 171 – 24 T);
Asphalt for Use in Damp-proofing and Waterproofing Below Ground Level (D 40 – 24 T);
Asphalt for Use in Damp-proofing and Waterproofing Above Ground Level (D 144 – 24 T);
Primer for Use with Asphalt in Damp-proofing and Waterproofing Below and Above Ground Level (D 41 – 22 T);
High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (D 42 – 24 T);
High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level (D 145 – 24 T);
High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (D 200 – 24 T);

High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level (D 201 – 24 T);

Creosote Oil for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing Below and Above Ground Level (D 43 – 22 T);

Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 173 – 23 T), revised as recommended by the committee;

Burlap Saturated with Bituminous Substances for Use in Waterproofing (D 174 – 23 T), revised as recommended by the committee.

The withdrawal of the Tentative Specifications for Felted Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 172 – 23 T) was approved.

On motion, the report of the committee was adopted.

A paper entitled "Machine for the Determination of the Pliability of Prepared Roofing and the Breaking Point of Bitumen," by Charles S. Reeve and Frank W. Yeager, was presented by Mr. Reeve.

The meeting adjourned till 8 P. M.

SEVENTH SESSION—THURSDAY, JUNE 25, 8 P. M.

On Research, Testing, Nomenclature and Specifications

President F. M. Farmer in the chair.

The report of Committee E-9 on Correlation of Research was presented by the chairman, H. F. Moore and, on motion, was adopted.

The report of Committee E-1 on Methods of Testing was presented by the chairman, J. A. Capp. The chairman then called on H. F. Moore to present the report of the Sub-Committee on Mechanical Testing of Metallic Materials. This report consisted of revisions in the present tentative methods of mechanical testing and in the submission of new methods for publication as information. On the recommendation of the committee, the revisions of the following Tentative Methods and Definitions of Terms were accepted, the Methods and Definitions of Terms being continued as tentative:

Tentative Methods of:

Tension Testing of Metallic Materials (E 8 – 24 T);

Compression Testing of Metallic Materials (E 9 – 24 T);

Brinell Hardness Testing of Metallic Materials (E 10 – 24 T).

Tentative Definitions of:

Terms Relating to Methods of Testing (E 6 – 24 T).

The Proposed Methods of Flexure Testing of Metallic Materials, the Proposed Methods of Torsion Testing of Metallic Materials and the Additional Method for Determination of Elastic Limit were accepted for publication as information as recommended by the com mittee.

The chairman then called on F. G. Breyer to present the report of the Sub-Committee on Classification of Material According to Size. This sub-committee had prepared proposed Tentative Specifications for Sieves for Testing Purposes which, on recommendation of the committee, were accepted for publication as tentative. The report of the committee was discussed and, on motion, was adopted, together with the report on Determination of Water, appended to the report.

The report of Committee E-8 on Nomenclature and Definitions was presented by the chairman, Cloyd M. Chapman. The proposed Definitions of Terms Relating to Specific Gravity and the proposed Definition for the Term Screen (Sieve) were accepted for publication as tentative as recommended by the committee. The revision of the Tentative Definition for the Term Sand (C 58 - 24 T) was approved, the definition being continued as tentative. On motion, the report of the committee was adopted.

Past-President J. A. Capp here took the chair.

A paper entitled "The Purchase of Materials on Specification," by Dean Harvey, was presented by the author and discussed.

EIGHTH SESSION—FRIDAY, JUNE 26, 9.30 A. M.

On Paints, Textiles, Petroleum Products and Insulating Materials

(Held Simultaneously with the Ninth Session)

Past-President J. A. Capp in the chair.

The report of Committee D-1 on Preservative Coatings for Structural Materials, in the absence of the chairman, Allen Rogers, was presented by the secretary, R. L. Hallett. The report as preprinted contained proposed Specifications for Toxic Ingredients in Anti-Fouling Paints. These specifications were withdrawn by the committee. The committee had also recommended revisions in the wording of the tentative definitions of the terms "elasticity" and "toughness." These revisions were likewise withdrawn by the committee. The proposed revisions of the Tentative Specifications for Raw Tung Oil (D 12 - 23 T) and of the Tentative Specifications for Iron Oxide and Iron Hydroxide (D 84 - 24 T) were accepted, the specifications being continued as tentative.

The following new tentative standards were accepted for publication as tentative as recommended by the committee:

Tentative Methods of:

Routine Analysis of White Linseed-Oil Paints;
Testing Shellac Varnish.

Tentative Specifications for:

Dry Bleached Shellac;
Lithopone;
Lampblack;
Bone Black;
Chrome Yellow;
Pure Chrome Green;
Reduced Chrome Green.

On recommendation of the committee the following three tentative methods were referred to letter ballot of the Society for adoption as standard:

Tentative Methods of:

Routine Analysis of Titanium Pigments (D 186 – 24 T);
Testing Oleo-Resinous Varnishes (D 154 – 24 T);
Test for Wax in Shellac (D 29 – 24 T).

The proposed revisions of the Standard Specifications for Turpentine (D 13 – 24) and of the Standard Definitions of the Terms Relating to Paint Specifications (D 16 – 24 [illegible] latter consisting of the addition of a definition for the term "screen (sieve)," were, on recommendation of the committee, accepted for publication as tentative. On motion, the report of the committee was adopted.

A paper entitled "Tests for Hardness, Gloss, Color, and Leveling of Varnishes," by A. H. Pfund, was presented by Cole Coolidge and discussed.

A paper entitled "A Recording Drying-Time Meter for Varnishes and Similar Materials," by J. McE. Sanderson, was presented by the author.

The report of Committee D-13 on Textile Materials was presented by the chairman, W. F. Edwards. The following tentative specifications were referred to letter ballot of the Society for advancement to standard as recommended by the committee:

Tentative Specifications for:

Tolerances for Hose Ducks and Belt Ducks (D 181 – 24 T);
Tolerances and Test Methods for Electrical Cotton Yarns (D 203 – 24 T), revised as recommended by the committee;
Imperfections and Tolerances for Square-Woven Tire Fabrics (D 122 – 23 T), revised as recommended by the committee;

Imperfections and Tolerances for Cord Tire Fabrics (D 179 - 23 T), revised as recommended by the committee;
Specifications and Methods of Test for Osnaburg Cement Sacks (D 205 - 24 T), revised as recommended by the committee.

These specifications contain a number of definitions of terms. These have been referred to letter ballot in Committee E-8 on Nomenclature and Definitions and have been approved by that committee with the reservation that a note be added to the definition for "Direction of Twist" appearing in the Specifications for Tolerances and Test Methods for Electrical Cotton Yarns (D 203 - 24 T) to read as follows:

> "The definition as here given does not agree with the definition of direction of twist of cotton sewing thread as appearing in the Tentative Specifications for Tolerances and Test Methods for Cotton Sewing Threads (D 204 - 24 T) which definition defines 'direction of twist' so that right-hand twist in the case of sewing thread corresponds to left-hand twist in the case of electrical cotton yarns, as defined herein. An attempt is being made to harmonize these two conceptions of 'direction of twist.'"

The proposed revisions of the Tentative Specifications for Tolerances and Test Methods for Cotton Yarns (D 180 - 24 T), for Tolerances and Test Methods for Cotton Sewing Threads (D 204 - 24 T), and for Tentative Specifications for Textile Testing Machines (D 76 - 22 T), recommended by the committee, were accepted, the specifications being continued as tentative.

On recommendation of the committee, the following new tentative standards were accepted for publication as tentative:

Tentative Specifications for Tolerances for Numbered Duck;
Tentative Specifications for Tolerances and Test Methods for Knit Goods;
Tentative Methods of Testing Grease Wool and Allied Fibers for Scoured Content.

On motion, the report of the committee was adopted.

A paper entitled "The Design of a Research Laboratory for a Textile Manufacturing Plant," by George B. Haven, was presented by the author and discussed.

The report of Committee D-15 on Thermometers, in the absence of the chairman, W. H. Fulweiler, was presented by E. N. Hurlburt. On recommendation of the committee the following three specifications were referred to letter ballot of the Society for advancement to standard:

Tentative Specifications for:

A.S.T.M. Partial-Immersion Thermometer for General Use, −20 to +150° C., 0 to +300° F. (D 182 – 23 T);

A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +300° C., +20 to +580° F. (D 183 – 23 T);

A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +400° C., +20 to +760° F. (D 184 – 23 T).

The committee presented Specifications for A.S.T.M. Low-Cloud and Pour Test Thermometers −60 to +20° C., −70 to +70° F., which specifications were being incorporated by Committee D-2 on Petroleum Products and Lubricants in its Tentative Methods of Test for Cloud and Pour Points of Petroleum Products (D 97 – 23 T). On motion, the report of the committee was adopted.

The report of Committee D-2 on Petroleum Products and Lubricants, in the absence of the chairman, C. P. Van Gundy, was presented by the vice-chairman, K. G. Mackenzie. A number of editorial changes were made in this report by the committee, principally in the Proposed Method for Determination of Oil in Wax, presented as information by Sub-Committee III on Paraffin Wax and in the proposed Tentative Method of Test for Penetration of Cup and Railroad Greases. Some additional changes were proposed in the Tentative Methods of Testing Gas Oils (D 158 – 23 T). These for the most part were rearrangements of the material appearing in the report as preprinted. The committee, however, changed its recommendation in reference to the proposed Abridged Volume Correction Table for Petroleum Oils. The report as preprinted recommended that the Table be accepted for publication as tentative. The committee now recommended that the Table be adopted immediately as standard. It is an abridgement of the tables contained in *Circular 154* of the U. S. Bureau of Standards. There is, at the present time, a great deal of confusion in the petroleum industry as to what abridged form shall be used. The American Petroleum Institute, as representing the petroleum industry, unanimously agreed on the form of abridged table now being recommended by Committee D-2. It was felt that it would be most desirable if the table should be adopted as standard by the A.S.T.M. There is no possibility of revision since the Table is merely a compilation of figures prepared by the Bureau of Standards. There would accordingly be no point to having it printed as tentative for the elicitation of criticism and suggestions. The committee accordingly asked for a nine-tenths vote of the meeting to have the Table referred to letter ballot of the Society for adoption as standard. This recommendation of the committee was unanimously approved.

The following proposed new tentative standards were accepted for publication as tentative as recommended by the committee:

Tentative Methods of:

Test for Penetration of Cup and Railroad Greases;
Test for Color of Petrolatum by Means of the Union Colorimeter;
Test for Neutralization Number of Petroleum Products and Lubricants;
Test for Burning Quality of Long-Time Burning Oil for Railway Use;
Test for Distillation of Natural Gas Gasoline.

The Method of Test for Neutralization Number of Petroleum Products and Lubricants listed above was in effect a revision of the existing Method of Test for Free Acid in Lubricants (D 188 - 24 T) and this tentative method was accordingly withdrawn.

The revisions recommended by the committee in the following tentative methods were accepted, the methods being continued as tentative:

Tentative Methods of:

Test for Viscosity of Petroleum Products and Lubricants (D 88 - 24 T);
Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 - 24 T);
Test for Cloud and Pour Points of Petroleum Products (D 97 - 23 T);
Testing Gas Oils (D 188 - 24 T).

On motion, the report of the committee was adopted.

At the request of the chairman, G. W. Thompson now assumed the chair.

The report of Committee D-9 on Electrical Insulating Materials was presented by its chairman, F. M. Farmer. In addition to the revisions in the tentative standards given in the report as preprinted, the committee recommended the withdrawal of Sections 13 to 19, inclusive, covering the description for flash-over voltage tests in the Tentative Method of Testing Electrical Insulating Materials for Voltage Effects at Radio Frequencies (D 175 - 23 T). This recommendation was approved, the Method being continued as tentative.

The revisions in the following Tentative Methods, recommended by the committee, were accepted, the Methods being continued as tentative:

Tentative Methods of:

Testing Insulating Varnishes (D 115 – 24 T);
Testing Cable Splicing and Pothead Compounds (D 176 – 24 T);
Testing Untreated Insulating Paper (D 202 – 24 T).

The revisions of the Standard Methods of Testing Molded Insulating Materials (D 48 – 24) recommended by the committee were accepted for publication as tentative.

On recommendation of the committee, the proposed Tentative Methods of Testing Laminated Sheet Insulating Materials were accepted for publication as tentative. On motion, the report of the committee was adopted together with the appendix on "Life Test for Transformer Oils" by E. A. Snyder and D. C. Cox, submitted by the committee for publication as information.

NINTH SESSION—FRIDAY, JUNE 26, 9.30 A. M.

On Cement, Lime and Gypsum

(Held Simultaneously with the Eighth Session)

Past-President George S. Webster in the chair.

The report of Committee C-1 on Cement, in the absence of the chairman, R. S. Greenman, was presented by the vice-chairman, W. M. Kinney. The report as preprinted contained revisions of the Standard Specifications and Tests for Portland Cement (C 9 – 21) and of the Standard Specifications for Natural Cement (C 10 – 09), these revisions being recommended for publication as tentative. The committee now recommended that the revisions be merely published as information. This recommendation was approved. On motion, the report of the committee was adopted together with the Manual of Cement Testing, this manual being intended to supplement the Standard Specifications and Tests for Portland Cement (C 9 – 21) and to be distributed with those specifications.

The following papers were presented by the authors and discussed:

"Requirements of Cement for Modern Highway Construction" by A. T. Goldbeck;

"A Study of Temperatures in High-Alumina Cement and Methods of Curing," by H. S. Mattimore.

The report of Committee C-7 on Lime, in the absence of the chairman, H. C. Berry, was presented by the vice-chairman, E. E. Eakins. On recommendation of the committee, the Tentative Specifications for Quicklime and Hydrated Lime for Use in the Cooking of Rags for the Manufacture of Paper (C 45 - 24 T) were referred to letter ballot of the Society for adoption as standard.

The revisions of the Standard Specifications for Hydrated Lime for Structural Purposes (C 6 - 24) recommended by the committee were accepted for publication as tentative.

The proposed revisions of the following Tentative Standards were accepted, with some minor editorial changes in the revisions of Specifications C 53 - 24 T and C 54 - 24 T, for publication as tentative, the specifications and methods of test being continued as tentative.

Tentative Specifications for Quicklime and Hydrated Lime for Use in the Manufacture of Sulfite Pulp (D 46 - 24 T);
Tentative Specifications for Quicklime for Use in Water Treatment (C 53 - 24 T);
Tentative Specifications for Hydrated Lime for Use in Water Treatment (C 54 - 24 T);
Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime (C 25 - 22 T).

On motion, the report of the committee was adopted.

The report of Committee C-11 on Gypsum was presented by the chairman, W. E. Emley. The following specifications, revised as recommended by the committee, were referred to letter ballot of the Society, for advancement to standard:

Tentative Specifications for:
Gypsum (C 22 - 24 T), revised as recommended by the committee;
Gypsum Plastering Sand (C 35 - 24 T), revised as recommended by the committee;
Gypsum Wall Board (C 36 - 24 T), revised as recommended by the committee;
Gypsum Plaster Board (C 37 - 22 T), revised as recommended by the committee;
Gypsum Partition Tile or Block (C 52 - 24 T), revised as recommended by the committee.

The proposed revisions of the Standard Methods of Testing Gypsum and Gypsum Products (C 26 - 23), recommended by the committee, were accepted for publication as tentative. The report was, on motion, adopted.

The revisions in the Tentative Definitions of Terms Relating to the Gypsum Industry (C 11 - 24 T), recommended by the committee, were accepted, the Definitions being continued as tentative.

A paper entitled "The Fire Resistance of Gypsum Partitions," by S. H. Ingberg, was presented by the author and discussed.

The meeting then adjourned till 8 P. M.

TENTH SESSION—FRIDAY, JUNE 26, 8 P. M.

On Concrete

President F. M. Farmer in the chair.

The report of Committee C-9 on Concrete and Concrete Aggregates, in the absence of the chairman, A. T. Goldbeck, was presented by the vice-chairman, Cloyd M. Chapman. On recommendation of the committee, the Tentative Methods of Making Compression Tests of Concrete (C 39 - 21 T) were referred to letter ballot of the Society for advancement to standard.

The proposed revisions of the Tentative Methods of Securing Specimens of Hardened Concrete from the Structure (C 42 - 21 T), on recommendation of the committee, were accepted, the Methods being continued as tentative. On motion, the report of the committee was adopted together with two appendices, the first one being a "Digest of Literature on Methods of Making Flexure and Tension Tests of Concrete," by Duff A. Abrams, and the second a form for "Concrete Aggregate Material Investigation Report."

The winners of the Golf and Tennis Tournaments were then announced and the prizes awarded by E. D. Boyer on behalf of the Entertainment Committee. The A.S.T.M. championship golf cup and medal for the low score were won by E. W. McMullen of Kenosha, Wis. The A.S.T.M. championship tennis cup and medal were won by R. J. Shoemaker of Chicago, Ill.

Other prize winners in golf and tennis were: W. B. Price, Herbert Spencer, J. E. Lockwood, D. W. Mulford, J. G. Detwiler, C. T. Ripley, A. Orr, E. D. Boyer, H. W. Bates, M. H. Ulman, H. S. Mattimore, R. H. Catlett, and R. P. Anderson.

The following five papers were presented and discussed.

"A New Test for Consistency of Paving Concrete," by F. H. Jackson and George Werner—presented by Mr. Jackson.

"Effect of Size and Shape of Test Specimen on Compressive Strength of Concrete," by H. F. Gonnerman—presented by the author.

"Studies of Bond Between Concrete and Steel," by D. A. Abrams—presented by the author.

"The Significance of the Common Test Methods for Determining the Strength of Mortars," by John W. Gowen and H. Walter Leavitt—presented by Mr. Leavitt.

"Analytical Properties of Set and Hardened Mortars," by E. E. Butterfield—presented by the author.

There being no further business, the Chair declared the Twenty-eighth annual meeting adjourned *sine die.*

American Society for Testing Materials

ORGANIZED IN 1898.
INCORPORATED IN 1902.

PROCEEDINGS

PART I

The Society is not responsible, as a body, for the statements and opinions advanced in this publication.

PROMOTION OF KNOWLEDGE OF ENGINEERING MATERIALS

Annual Address by the President, F. M. Farmer, June 24, 1925

Custom in our Society prescribes that the retiring President shall deliver an address, and, in accordance with that unwritten mandate, I purpose directing your attention for a brief period to Society affairs.

The charter of the American Society for Testing Materials states that its objects are "the Promotion of Knowledge of Materials of Engineering and the Standardization of Specifications and Methods of Testing." One authoritative definition of promotion is "to contribute to the development, establishment, increase or influence of." Certainly the standardization of specifications and methods of testing does all of these things for engineering materials. So I should say that the latter portion of our charter statement could be included in the first part and that the broad fundamental purpose of the Society is the "Promotion of Knowledge of Materials of Engineering." It is perhaps somewhat unfortunate that our name implies so restricted a field as that of merely testing materials, but the significance of a name lies not so much in what it is as in what it stands for.

All of the many technical and engineering organizations of the country have promotion of knowledge in their respective fields as a direct or indirect object. The American Society for Testing Materials, however, occupies a unique position. In the first place, it serves all branches of industry since all use engineering materials to a greater or less extent, either directly or indirectly. In the second place, it not only contributes to the general fund of scientific and technical knowledge of engineering materials, but it renders a definite, concrete service through the development and standardization of specifications and methods of testing—activities which have a direct economic value to industry and therefore to the public.

Purpose of Promotion of Knowledge of Engineering Materials:

What is our purpose in promoting knowledge of engineering materials? We are an organization which is a part of, is supported by, and is contributing to the industry of this country. Industry is very materialistic and our object, while it may be an ideal, must, if we are to be of maximum service, be a practical one.

Our purpose can, I believe, be best stated thus: to increase the efficiency and effectiveness of materials of engineering. And so I should say that we are organized for the purpose of promoting knowledge of engineering materials in order that they may be more efficiently and more effectively used in industry.

Knowledge can be promoted by the Society in three ways: by developing and publishing information obtained through research carried on by the Society itself, by publishing information developed by its members individually, and by extending the usefulness of existing information. Through research the circle of knowledge is widened, while through education and publicity the contents of the circle are made available to a larger proportion of humanity and thus become more useful.

Promotion Through Research:

The Society is contributing greatly to the promotion of knowledge of engineering materials through active research work being carried on by its committees and by its members individually. I need only refer to the list of the more notable examples of such work now in progress under our committees which is given in the report of Committee E-9 on Correlation of Research. This work involves no expense to the Society except in connection with the publication of the results obtained. However, President Burgess, in his address of two years ago, pointed out that we as a body should be more actively engaged in research work on some of the many fundamental problems in the field of engineering materials. I heartily agree with this view.

The lack of financial resources has prevented the Society as a body from undertaking research work on its own account in its own particular field. Yet millions of dollars are being expended annually in research by private industrial interests. Many of the industries are doing cooperative research work on an extensive scale in their respective fields and a fund of $20,000,000 for research work by Engineering Foundation on engineering problems has been proposed. Is it too much to hope that in time this Society will have a research fund so that it will be in position more effectively to discharge this part of the obligation which it has assumed?

Basic research on engineering materials should be carried on cooperatively by all interests concerned. This not only distributes the costs more equitably, but permits drafting the best available talent which is familiar with both the consumer and the producer sides of the technical and commercial problems involved. This Society provides just these conditions and there is no more appropriate channel through which research work on the fundamental problems of engineering materials can be carried on.

Promotion Through Publication of New Knowledge:

The Society's most valuable contribution to the knowledge of engineering materials has so far been its standardization work—work which has earned for the Society an enviable reputation. But as standards are established in the more important classes of engineering materials, the opportunities for further useful service in this direction must diminish. True, progress and changes in the industrial arts with increase in knowledge of materials and methods of using them will necessitate revision of these standards from time to time in order to maintain them at a maximum value to industry. Furthermore, we shall always be developing standards in new fields as well as new standards in old fields. Nevertheless I believe we are approaching the time when our service to industry in contributing new information on engineering materials will be relatively as great as that rendered in standardization work.

What is the situation in regard to this phase of the Society's objects? I have already indicated that the Society is not in a position to undertake the development of new knowledge through research work of its own. Not only is this true, but the Society has grown to the point where it cannot even publish the results of all of the research and investigation being done by its members individually. The condition that confronted our Papers and Publications Committee this year shows very clearly our situation.

Last year our Proceedings contained 2300 pages, several hundred pages more than the average of the last few years. The report of the Executive Committee points out that a large part of this excess was due to certain symposiums and special reports amounting to 647 pages.

Due to the publication of this large amount of material we exceeded our budget allowance for printing by about $4000. Our Executive Committee felt, however, that these symposiums and reports, containing, as they did, a vast amount of data obtained at great cost but given freely to the Society, should be published despite the budget. The immediate importance, for example, of new knowledge of the properties of materials at high temperatures will be recognized by everyone, particularly in its application to such industries, for example, as that of electrical energy production and petroleum production. It is generally recognized that one of the principal factors which has permitted the reduction in costs of electrical energy to the public during the last few years, while the cost of most other commodities has risen, is the greater efficiency obtained by using higher steam pressures and temperatures. Similarly, in the petroleum industry, the demand for more gasoline has been met by the use of higher temperatures and pressures in the cracking processes. But, in the light of our present knowledge, the safe limit of use of the materials employed in boilers, piping and retorts has been about reached. Since further progress must be very largely in the direction of higher temperatures, it follows that we must know more about the properties of these materials at high temperatures.

Similarly, knowledge of the alloys suitable for industrial applications is of the greatest economic importance because advance in many technological fields is dependent upon the use of alloys. Here again everyone will recognize the value of knowledge which contributes, for example, to the solution of the corrosion problem—that source of waste which has been stated to be costing the people of the United States $300,000,000 per year. And so, while all of these various problems are of immediate concern to industry, progress toward their solution is of very direct economic importance to the general public.

As a consequence of publishing this extra material last year, our budget allowance this year has had to be reduced and a number of papers on timely subjects have had to be refused, and the "blue pencil" very energetically employed. I mentally hear comments that this may not be an unmixed blessing and that the "blue pencil" has not been used enough in the past! There will always be differences of opinion on what material should be accepted for publication and how much it should be condensed. Nevertheless the fact remains that we cannot begin to publish all of the material that is available—material which any committee on papers would agree should be accepted. This is due to the growth of the Society and the increasing recognition of the

Society as the most appropriate channel for the publication of results of research and investigation on engineering materials. And what are we to do about it? Shall we publish only what our present income will permit, and therefore limit our usefulness, or shall we increase our income and endeavor to fulfill the duty which we have imposed upon ourselves? There is only one answer—if anything is worth doing at all, it is worth providing the necessary means for doing it well.

Promotion Through Greater Use of Existing Knowledge:

I wish to place special emphasis not on the promotion of knowledge by the development of new information through active research or the publication of results thereof, but rather on that other method of promoting knowledge, namely, increasing the usefulness of the knowledge we already have. As an organization of technologists and engineers whose creed is the efficient application of science to industry, we should see to it that the product of our activity as an organization is efficiently applied and utilized to the greatest extent not only by our own members but by the public and industry in general.

Good engineering is just as important in the preparation of specifications for engineering materials as it is in the application of such materials. The four hundred standards which have been developed by the Society presumably have been prepared on that basis with due regard for the economic and technical requirements of both consumer and producer interests. Having made it our first duty to see to it that our standards are as nearly right as possible, then we should see to it that they are made as useful as possible, for thus do we accomplish our primary purpose, namely, the more effective, more efficient and more economic use of materials of engineering.

How can we increase our usefulness? One means is through technical and particularly general press publicity. We have, I believe, been too self-centered, and have considered that our work concerned primarily only our own members. Also, perhaps we have felt that anything savoring of advertising was unethical for a national technical organization. Whatever may have been the reason, the fact is that we have not made our work known as much as it should be and our usefulness is not what it might be. One of the most effective agencies for promotion of knowledge is publicity and we should use all dignified means to advertise our work to industry and to the public, not for the purpose of exploiting the Society as such but to make the results of the large amount of time and energy expended in our activities known to a wider circle and thus increase their usefulness.

Another means of promoting our usefulness is through our members themselves. Our own members can greatly aid in giving publicity to A.S.T.M. standards by giving credit to the Society whenever they use A.S.T.M. standards in whole or in part in their own specifications or other standards. It is natural perhaps for an organization using specifications to desire to retain its own individuality in connection therewith. But where A.S.T.M. standards have been used, indicating the source will not only strengthen the specifications but will help in gaining prestige for the work of the Society which you are supporting. Our standards are used widely and in various degrees. Sometimes they are quoted bodily and verbatim, while in other cases only particular sections are quoted. Again, a whole specification or certain sections may be used with certain modifications. In any case, the material used, be it a whole specification or method of test or only a portion, could be followed by an appropriate reference in parenthesis such as "A.S.T.M. Standard C 9 - 21" or "Section 5, A.S.T.M. Tentative Standard A 92 - 24 T," or "Based on A.S.T.M. Standard D 48 - 24." The specifications will then not only have a more substantial basis, but the manufacturer or vendor will recognize and appreciate the effort toward simplification and standardization.

The technical schools present a large opportunity for increasing our usefulness. We should make available to instructors dealing with materials of engineering our long experience in the building of specifications. Lecture material can be supplied which would include such topics as the purpose of specifications; the proper criterion; kinds; process and technique of constructing specifications; importance of standard methods of testing; application and use; importance of inspection, etc. Alumni who are active in our Society could very properly give such lectures together with talks on the work of the Society. This would not only be contributing knowledge to the student, but we would be extending our sphere of influence because the student of to-day is the man of responsibility in industry of to-morrow.

The publication by the Society of a technical journal has been discussed by our Executive Committee several times in the past and has not been considered advisable. It is obviously too large a problem with too many angles to be properly discussed here, but a journal has several advantages, not the least of which is the opportunity afforded for greater usefulness for the Society through the greater and more frequent publicity which would thus be obtained. And I believe that a publication policy could be worked out in which a journal would replace a part, if not all, of that volume of our Proceedings which contains the papers without adding greatly to our present publication costs.

Other directions which may be mentioned and in which our usefulness can be increased include provision for student membership at very nominal dues, broader distribution of tentative standards so that a wider range of criticism and comment can be obtained, and more specific attention to the needs of the smaller consumer—as was so very well pointed out by T. H. Wiggin in his paper presented at the last annual meeting.[1]

Cost of Society's Work and its Value to Industry:

May I now turn to that very practical question, the cost and value to industry of our activities in promoting knowledge of engineering materials? The cost of the 2500 trade and technical organizations said to be functioning in this country and the value of their accomplishments is not an infrequent topic of discussion among industrial executives. This is a question which we in the A.S.T.M. should face fairly and squarely for, as a society of engineers and technologists dealing with practical affairs, we should be able to justify our cost to industry.

The direct cost of carrying on the Society's work through the cash disbursements from the Treasurer's office is about $80,000 per year. However, by far the larger portion of the total cost of the Society to industry is the indirect cost contributed by our committees. An analysis of the activities of our standing committees based on estimates supplied by committee chairmen for the Society year 1923-24 indicates the following:

	Committee Meetings		Committee Activities other than Meetings		Total Estimated Cost
	Men-days	Estimated Cost	Men-days	Estimated Cost	
Metals Committees	900	$50,000	1450	$35,000	$85,000
Non-Metals Committees	1450	75,000	2350	70,000	145,000
Administrative Committees	125	6,000	....	.....	6,000
Total	2475	$131,000	3800	$105,000	$236,000

While these figures are necessarily only approximations, it can be concluded that the total cost of the Society to industry is of the order of $300,000 to $325,000 per year—a very reasonable figure considering that we have over forty committees actively functioning and devoting over 6000 men-days to our work. When we examine the financial statements of some of the other large national technical organizations of the country and add thereto the cost of their committee activities, we find this sum very modest indeed, particularly when it is considered that our Society shows concrete and permanent tangible results in return to industry for its support. Incidentally, it may be noted that the value of the annual business done by the industries receiving the benefit of our work is of the order of thirty to thirty-five billions of dollars.

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[1] T. H. Wiggin, "How Shall the Benefits of A.S.T.M. Standardization be Secured to the Small User?" *Proceedings*, Am. Soc. Testing Mats., Vol. **24**, Part II, p. 1097 (1924).

Now as to the value received by industry in return for this expenditure, it is of course impossible to assign even an approximate figure in dollars. But use implies value and many instances of use of our standards can be cited. The following are typical:

1. In codes of various kinds, such as the building codes of several cities, government construction codes, the A.S.M.E. boiler code and the Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete.
2. Directly or indirectly in all large constructions involving steel and cement. Practically all bridge and building steel is in accordance with A.S.T.M. standards.
3. Directly or indirectly by many railroads. Locomotive builders use A.S.T.M. standards to a very considerable extent, especially on export work.
4. A.S.T.M. methods of test are widely used in industries where as yet there are no standard specifications, such as the petroleum industry, the electrical insulating material industry and highway construction.
5. In the textile field, the pioneer standardization work of Committee D-13 on Textile Materials is putting the buying and selling of textiles on a specification basis with great economic and other advantages to all concerned.
6. Finally, I would cite the indirect returns received by the committee members themselves. There can be no question that the indirect value to industry of the educational advantages to the 1000 men on our committees is several times that part of our total cost which they contribute.

Additional Income:

It has been pointed out that in certain important directions our usefulness is limited by the lack of financial resources. The principal one of these is our inability to publish all of the material offered to us and which is a distinct addition to our knowledge of materials of engineering. Our recent experience shows that we should have a greater annual income if our present expenditures are to be made more effective and the Society more useful to industry.

Several ways of increasing the Society's income will suggest themselves, such as increasing the dues, greatly increasing the membership and the establishment of a permanent fund, the annual income of which would be available for the publication of the results of research work.

I do not believe that the regular annual dues should be increased. The present dues, $15, compare favorably with those of other technical organizations. Furthermore, we want to get the maximum distribution of our publications and therefore we should make the conditions of membership for individuals as favorable as practicable.

Obviously, growth in membership means increase in income, but the surplus per member over the carrying charges is not sufficient to meet our needs. Nor should we curtail the amount of material supplied to members in order to increase this margin, for by so doing we defeat our object. But, on the other hand, neither should we give members too much more than they pay for, because nothing is valued so lightly as that which costs nothing.

Industry, both consuming and producing, profits directly by the work of this organization to an extent far in excess of the present costs. That being the case, why should not all industry contribute directly the additional revenue necessary to make our work more effective? The establishment of a permanent fund by industry has been suggested, but I do not believe that this is the best plan for several reasons, the principal one of which is perhaps a psychological one. The establishment of funds of various kinds was found to be so easy during and immediately following the war that the habit seems to have become fixed, and we have had so many "drives" for funds for every conceivable purpose, that that method of raising money is not popular. Another important reason is that such a method is not strictly fair because conditions in industry are constantly changing. It is questionable whether industries of to-day should be asked to supply funds the income from which will be used for the benefit of industries of to-morrow. I feel that our appeal should be on a business basis and that we should seek increased financial support from industry only where and while such support is considered a profitable expenditure.

The best method of meeting the problem is, I believe, the establishment of a special class of membership wherein the dues would not be limited to $15 per year. It is obvious that business organizations should bear a larger proportion of the cost of operating the Society than individuals and that they should pay more than $15 per year.

It has been suggested that a class of members, known as Sustaining Members, be established which, while open to all, would be expected to be availed of principally by industrial and commercial organizations. The amount of dues should be entirely optional with each member except that there should be a maximum limit. Thus the contribution to the Society's work by each such member would presumably be dependent upon the value of our work to that member and that, to my mind, is the great advantage of the plan. It is hardly necessary to point out that provision should be made to safeguard the traditional policy of the Society with reference to producer and consumer influence—a policy which has contributed so greatly to its success. One hundred such members contributing annually an average of $100 each would increase our income $10,000—a sum which would solve our immediate publication problem quite satisfactorily. An additional income of $25,000 would be only about eight per cent of the cost of the Society's work, but such a sum would increase the effectiveness of our present expenditures in far greater proportion.

Other Problems:

One of the privileges enjoyed by the President in his annual address is that he may depart from his text without being more or less gently admonished by the presiding officer. I cannot, therefore, refrain from touching on two matters which have been the subject of discussion in our ranks. I refer to the consumer *versus* producer problem and to the relation of our standardization work to that of other bodies.

One of the most important policies which the Society has always followed is that producers shall have an equal voice with consumers in the formulation of standards, but there are occasional expressions of opinion that the producer interests on this or that committee are in control of the situation. Now what are the facts? In the first place, it is obvious that in general the producer will be more actively interested in standardization work because its economic value can be more directly translated by him into dollars and cents. Furthermore, he is of necessity likely to be better equipped with technical talent, with data, with knowledge and with facilities for acquiring further knowledge than are the consuming interests. This is so not only because it is necessary for commercial success, particularly in a competitive field, but the producer interests in general are concentrated in far fewer and much larger units with greater resources than are the consuming interests. Again, in many branches of industry the producers work cooperatively and can present a coordinated front, so to speak, on specification questions, while the various classes of consumers are not so generally organized in associations and frequently cannot agree among themselves as to what the requirements should be from the consumer's standpoint. And so while the voting strength in our committees may be balanced, the preponderance of real weight is undoubtedly frequently in favor of the producer. But this does not necessarily mean that in all such cases the producer interests take an improper advantage of the situation. Ethical standards in industry were never higher than they are to-day for the very practical reason that fair dealing pays the highest dividends. Specifications, for example, which have been developed with the best interests in view of *all concerned* are, in the long run, the best for the interests of the producer. But where the situation is not satisfactory, the fault usually is, I feel, that of the consumer interests.

In such cases what is the remedy? Greater activity on the part of, and more coordination among, the consumers on the committee. This Society, of all bodies doing standardization work, offers the greatest opportunity to consumers, but the consumer interests, large and small, must take advantage of that opportunity if results entirely satisfactory to them are to be obtained. In this connection I may mention the criticism of a similar tenor occasionally received from a large consumer or a group of consumers not represented on the committee involved and perhaps not even a member of the Society. The remedy in this case is obvious. Let the complaining witness profit by his opportunity to take active part in the development of the standards and make his criticism during their formulation rather than after their completion.

Standardization work by other bodies has increased greatly during the past few years and these activities are sometimes cited as detracting from the usefulness of our Society. While the development of these other activities may sometimes appear to indicate a trend away from rather than toward simplification in standards, I do not believe this situation can be avoided or that there is not still plenty of opportunity for usefulness in standardization work. We cannot expect to do all of the standardization in materials, and when a special branch of industry, like the automotive industry, for example, grows sufficiently large, it is only logical that it should undertake the preparation of its own standards of various kinds, including specifications for engineering materials, so many of which are special in that particular field. We should cooperate with such bodies as much as possible, making available to them our experience and accomplished results and thus not only be useful but avoid the real danger, namely, duplication of work already done. One of the principal advantages of the American Engineering Standards Committee to this Society is the opportunity afforded for just such cooperation, which results in greater familiarity of other organizations with the work already accomplished by this Society, thus increasing our prestige and influence.

But there is ample opportunity to serve general industry, for all industry employs engineering materials to a greater or less extent. It is the multiplicity of specifications for engineering materials for general use that should be reduced in the interest of elimination of waste and economy in manufacture, distribution and use of such materials.

Conclusions:

In conclusion I venture to suggest that America's leadership in the industrial world is due not only to efficiency through mass production and the efficient use of men and materials, but to the application of knowledge made available through research and the cooperative work of scientists, technologists and engineers in industry. The American Society for Testing Materials is responsible for much of the research and cooperative work in the field of engineering materials and has therefore been a significant factor in our industrial progress. Continued effort is essential, however, if America is to maintain its position as international competition becomes keener and labor's share in the profits of industry becomes greater.

Many of the matters to which I have referred in this address are being studied by our Executive Committee and by a special committee on Promotion of Usefulness. When in due course definite recommendations are made to the membership, let us promptly determine to increase our contribution to the common cause of maintaining America's industrial supremacy by making our work still more useful to industry through more intensive promotion of knowledge of engineering materials by all of the various means at our disposal.

ANNUAL REPORT OF THE EXECUTIVE COMMITTEE

The Executive Committee offers the following report of general Society activities for the past year:

Membership:

Membership statistics for the past year are shown in the following table:

	1924 Annual Meeting	1925 Annual Meeting	Losses				Additions		Totals		
			Transfer	Resignation	Dropped	Death	Transfer	Election	Loss	Gain	Increase
Honorary Members	3	2	..	..	..	1	..	..	1	..	1[a]
Members	3387	3635	..	114	92	16	23	447	222	470	246
Junior Members	91	79	23	5	17	0	..	33	45	33	12[a]
Total	3481	3716	23	119	109	17	23	480	268	503	235

[a] Decrease.

The membership of the Society at the time of this annual meeting is 3716. The net increase in membership for the year now ending is 235 as compared with 241 for the year ending with the 1924 annual meeting and 210 for the year ending with the 1923 annual meeting. The number of new members elected during the year (480) compares very favorably with the records of the two preceding years and is due in large part to the cooperation of the standing committees in using their organized efforts to secure new members. What has been done to stimulate efforts along this line has been reported from time to time in the A.S.T.M. Bulletin. The interest of the standing committees and of the members individually has been very gratifying and the Executive Committee expresses its appreciation of the efforts that are being made along this important line.

The Society has suffered the loss by death of seventeen members. The names of the deceased members are:

Genji Araki
A. O. Backert
John B. Bird
W. P. Blair
Charles W. Burrows
D. T. Day
J. W. Dougherty
W. H. Harding
Gabriel Henry
W. F. Hillebrand
Olaf Hoff
E. V. Johnson
Jonathan R. Jones
H. R. Kimmel
J. B. Lober
Mansfield Merriman
W. T. Morgan

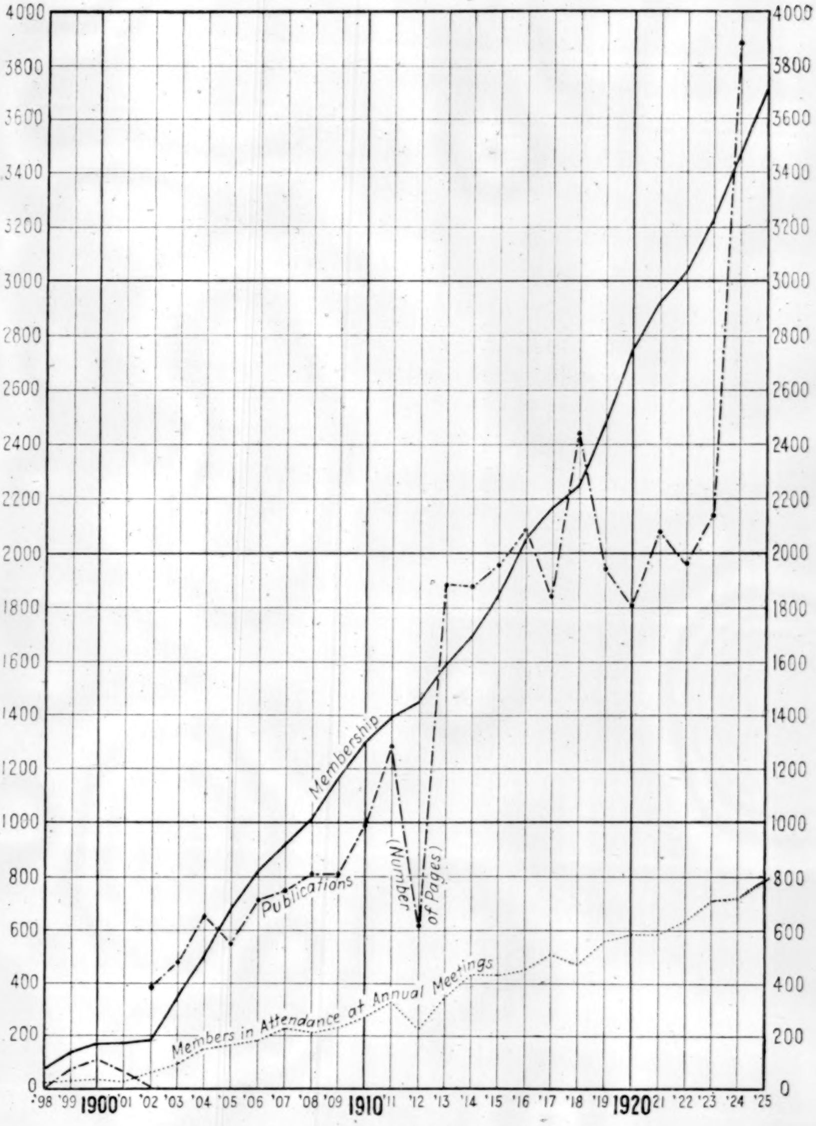

FIG. 1.—Membership and Publications Chart.

NOTE.—The initial curve for publications (1898–1902) refers to the publications of the American Sectinn of the International Association for Testing Materials. The marked drop for 1912 in the "publications" and "members in attendance at annual meeting" curves is due to the fact that the Sixth Congress of the International Association was held in New York in September, 1912, and that the activities of the American Society for that year were accordingly restricted to committee reports and administrative business.

Membership growth since the organization of the Society is shown in Fig. 1.

Standing Committees:

The standing committees of the Society have been quite active within the year. Since the last annual meeting two new committees have been organized, namely, E-9 on Correlation of Research and D-17 on Naval Stores, making a total of 43 standing committees. Of these committees, four are not presenting reports this year, five have submitted formal reports of progress to the Executive Committee, and reports from the remaining 34 are announced on the program of this meeting. These reports comprise approximately 600 pages and include recommendations for proposed revisions in 29 standards, adoption of 36 tentative standards as standard, acceptance of 45 proposed new standards as tentative, and proposed revisions in 37 tentative standards to be continued as tentative. From this it will be seen that the work of developing standard specifications and methods of test is progressing actively. There are at present 405 standards and tentative standards of the Society, and if the recommendations of the various committees are approved at this annual meeting, this figure will become 447, an increase of 42.

During the year, Committee C-10 on Hollow Building Tile advised the Executive Committee that its program of work on hollow tile of burned clay had been brought to such a degree of completion that the committee felt prepared to undertake the formulation of specifications for hollow tile of other materials and to make such changes in its membership and organization as would best enable it to function under its enlarged scope. The Executive Committee authorized the committee thus to enlarge its scope of activities and the title of the committee was changed to Committee C-10 on Hollow Masonry Building Units. In the proposed expansion of the committee's activities, the Executive Committee has ruled that the subject of hollow brick shall be assigned to Committee C-3 on Brick, the subject of lime tile or block to Committee C-7 on Lime and the subject of gypsum tile or block to Committee C-11 on Gypsum.

The Executive Committee has considered certain questions relating to the classification of members of standing committees submitted to it by Committee D-2 on Petroleum Products and Lubricants for decision. The Executive Committee has ruled that the present method of classification and counting of members of standing committees for the purpose of official Society records shall be continued; that any standing committee for its own records may classify its membership as to votes in accordance with the requirements of the Regulations Governing Standing Committees; and that, where these two classifications may differ, a note may be included in the Year Book at the committee's request giving the number of producer and non-producer voting memberships in accordance with such classification.

Committee D-2 also asked for purpose of committee membership classification what constitutes a "firm, company, corporation, laboratory or other institution" as used in the Regulations Governing Standing Committees, and whether only subsidiary companies should be considered with the parent company as one company or whether those companies of which the controlling stock is held by another company should also be considered as a part of that company. The Executive Committee has ruled that the interpretation of these terms as applied to committee membership shall be left to the respective standing committees to decide in the light of the particular conditions surrounding the industries with which they are associated, with the understanding that if any member of a committee is dissatisfied with a ruling on this subject by a standing committee, he shall have the right of appeal to the Executive Committee, similar to the right of appeal now granted in the Regulations Governing Standing Committees with respect to classification of members of committees into producers and non-producers.

As a result of consideration that has been given to the method of publishing the personnel of standing committees and their sub-committees in the Year Book, it is planned to give in parenthesis after the names of individual members of standing committees the names of the companies with which they are connected where this is applicable; and in printing the list of members of the sub-committees, to give only names of individuals, both in the case of individual memberships as at present and of the representatives of firms, companies, corporations, etc., holding membership as such on the committees.

During the year, the plan of group meetings of standing committees has been successfully continued. In October, 1924, nine of the metals committees held a three-day series of meetings at the Bureau of Standards in Washington, D. C., and in March of this year eleven metals committees and twelve non-metals committees held three-day group meetings in Philadelphia in successive weeks. The details of these meetings have been fully reported in the A.S.T.M. Bulletin. The March meetings were the first of such group meetings for the non-metals committees and, as in the case of the metals committees, the meetings were quite successful. The Executive Committee will continue to encourage such meetings when they can be arranged to suit the convenience of a number of the Society's committees. The Executive Committee further looks with favor upon the plan by which these meetings, which can probably be held in the fall and spring of each year, are alternated between the East and the West.

Publications:

The regular publications during 1924 were the Proceedings, Book of A.S.T.M. Standards and the Year Book. The comparative statement of the volume of regular publications during the past three years is as follows:

	1922, Pages	1923, Pages	1924, Pages
Proceedings	1614	1689	2306
Book of A.S.T.M. Standards	....	....	1230
Supplement to Book of Standards	54	126	....
Year Book (Membership List)	304	326	328
	1972	2141	3864

In addition, the Society published in 1924 the Book of A.S.T.M. Tentative Standards (763 pages), which is the only publication containing all of the tentative standards of the Society. It is furnished to members upon order at special prices, and the demand for it has steadily increased since its first appearance in 1917. This book brings the total number of pages of bound publications issued since the last annual meeting, but not including preprints and reprints, up to 4637 compared with 3000 for the preceding year.

The A.S.T.M. Bulletin has been issued at approximately quarterly intervals during the year, and four circulars to members have been printed. Reprints of several committee reports have been published for committee use and for sale, the principal among them being the report of Committee D-2 on Petroleum Products and Lubricants (80 pages) and the Joint Committee Report on Standard Specifications for Concrete and Reinforced Concrete (152 pages). In addition, the Symposium on Effect of Temperature Upon the Properties of Metals (184 pages) and the Symposium on Corrosion-Resistant, Heat-Resistant and Electrical-Resistance Alloys (269 pages), each complete with discussion, have been reprinted for sale. There has been and continues to be considerable demand for these several reprints.

It will be noted that the Proceedings for 1924 (2306 pages) are slightly over one-third larger in volume than the Proceedings for 1923 (1689 pages), which were the largest up to that time. This is due primarily to the fact that a number of important projects were developed and brought to the Society for publication last year. Of these the outstanding were two special reports from the Joint Committee on Investigation of the Effect of Phosphorus and Sulfur in Steel (90 pages), one involving a large number of photomicrographs with two large folding half-tone inserts; the report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete (84 pages); the Symposium on Effect of Temperature Upon the Properties of Metals (184 pages) held jointly with the American Society of Mechanical Engineers at Cleveland; and the Symposium on Corrosion-Resistant, Heat-Resistant and Electrical-Resistance Alloys (269 pages), including three expensive insert tables of data on these alloys. In addition, there were a number of voluminous papers on the program of the 1924 meeting, many of which included extensive tabular matter. The Executive Committee agreed with the Committee on Papers and Publications that the Society should not turn away this promising material that lay directly within its field. Its publication was accordingly authorized and the resulting deficit was met by a loan of $4000 secured by Liberty Loan Bonds as collateral. This loan has been repaid out of the current receipts for 1925, reducing by that amount the sum available for Proceedings for this year.

It should be pointed out that the sale of reprints of some of this additional material, principally the Joint Committee Report on Concrete and the two Symposiums, will in the next two or three years partly reimburse the Society for the deficit incurred.

Finances:

The annual statement of the finances of the Society follows in the form of the report of the auditors for the fiscal year January 1, 1924, to December 31, 1924.

The surplus on December 31, 1924, was $25,912.51 and includes $1600 life membership dues, $3672.90 for furniture and fixtures and $495.94 set aside as a sinking fund towards defraying the cost of the Index to Proceedings.

Referring to the table showing the financial condition of the Society at the close of the last five fiscal years, the drop in surplus from $37,133.03 in 1923 to $25,912.51 in 1924 is accounted for by the loan of $4000 previously mentioned and the sale of securities in the amount of $7200 included in the surplus for 1923, which were held in a sinking fund to meet the expense of publishing the 1924 Book of Standards.

The expenditure for rental of headquarters ($1382.83) is made up of $532.93 (the amount due in the fiscal year 1924 under the terms of

REPORT OF THE AUDITORS FOR THE FISCAL YEAR JANUARY 1, 1924, TO DECEMBER 31, 1924.

JOHN HEINS AND CO.
PUBLIC ACCOUNTANTS AND AUDITORS

PHILADELPHIA, January 7, 1925.

AMERICAN SOCIETY FOR TESTING MATERIALS,
MR. C. L. WARWICK, *Secretary-Treasurer*,
Philadelphia, Pa.

Dear Sirs:

We respectfully report that we have made an audit and examination of the books and accounts of your Society for the six months ended December 31, 1924, having previously made a similar audit and report for the preceding six months ended June 14, 1924, and at both audits found the accounts to be correct and to be in their usual excellent condition.

We submit balance sheet as of December 31, 1924, as also a statement of cash receipts and disbursements for the twelve months then ended.

We have verified the transactions set forth in the budget record for the last six months as also for the six months ended June 14, last, as reported in ours of June 23, last, and found the same to be correct.

We have verified by actual inspection and count the securities as listed on the Balance Sheet.

Respectfully submitted,
(Signed) JOHN HEINS AND CO.

BALANCE SHEET AS OF DECEMBER 31, 1924.

ASSETS.			
Cash		$282.06	
Investments:			
$13,200 U. S. Liberty Loan Bonds, 4th 4¼'s.	$12,685.03		
1,600 U. S. Liberty Loan Bonds, 4th 4¼'s, held for Life Membership Fund	1,525.95		
500 U. S. Liberty Loan Bonds, 4th 4¼'s, reserve for Index to Proceedings	495.94		
3,000 P. R. R. Equipment Bonds, 6's, due 1933	3,052.56		
2,000 Michigan Central Equipment Bonds, 6's, due 1932	2,085.96		
		19,845.44	
Accounts receivable:			
For Publications	$4,594.57		
Members for 1924 dues	2,230.85		
Members for binding	84.00		
		6,909.42	
Furniture and Fixtures		3,672.90	
			$30,709.82
LIABILITIES.			
Publications paid for in advance	$60.24		
Members dues paid in advance	734.07		
Members binding paid in advance	3.00		
Girard Trust Co. loan	4000.00		
		$4,797.31	

LIABILITIES (*Continued*) Brought Forward		$4,797.31	
Life Membership	$1,600.00		
Surplus	24,312.51		
		25,912.51	
			$30,709.82

MISCELLANEOUS FUNDS.

In addition to the above cash the Secretary-Treasurer has on hand the following funds:

Committee A-5 on Corrosion of Iron and Steel	$9,218.61	
Committee C-1 on Cement	1,648.43	
Committee C-9 on Concrete and Concrete Aggregates	610.69	
Committee C-10 on Hollow Building Tile	206.69	
Committee D-1 on Preservative Coatings	31.64	
Joint Committee on Investigation of Phosphorus and Sulfur in Steel	108.45	
		$11,824.51

RECEIPTS AND DISBURSEMENTS.

JANUARY 1, 1924, TO DECEMBER 31, 1924.

Cash on hand January 1, 1924			$5,139.72

RECEIPTS.

Current dues	$50,912.43		
Past dues	945.00		
Advance dues	719.19		
		$52,576.62	
Sale of Publications:			
Separate Standards	$3,587.22		
Book of Standards	4,338.07		
Book of Tentative Standards	6,213.45		
Proceedings	2,872.01		
Miscellaneous	3,503.55		
		20,514.30	
Binding (Members)		1,136.50	
Sale of right to reprint Standards		625.00	
Authors' reprints		58.05	
Sale of Certificates of Membership		40.00	
Interest on deposits and investments		2,398.54	
Accrued interest and premium		466.01	
Excess remittances		28.35	
Sale of reprints to cooperating societies, at cost		2,150.24	
Investments reserved for 1924 Book of Standards, matured or sold		9,677.44	
Girard Trust Company loan		4,000.00	
Contributions from members of standing committees to Entertainment Fund, Annual Meeting		495.00	
Seaview Golf Club charges, reimbursed by members		279.30	
Total receipts		$94,445.35	94,445.35
Carried forward			**$99,585.07**

Brought forward			$99,585.07

DISBURSEMENTS.

Publications:			
Year Book	$2,752.02		
Proceedings	27,528.82		
Book of Standards	11,673.37		
Book of Tentative Standards	2,482.67		
Preprints	3,682.84		
Miscellaneous	6,092.15		
		$54,211.87	
Salaries		21,746.80	
General Office Expenses		4,770.12	
Expenses, Standing Committees		1,797.27	
Expenses, Annual Meeting		2,542.51	
Expenses, Nominating Committee		55.66	
Traveling Expenses, Executive Committee		623.50	
Rent and Insurance, Storage Rooms		321.00	
Rent, Headquarters		1,382.83	
Headquarters' Maintenance		180.06	
Furniture and Fixtures		949.68	
Expenses, Enlargement of Headquarters		523.96	
Certificates of Membership		7.00	
American Engineering Standards Committee		1,513.72	
Annual Tables of Constants and Numerical Data		100.00	
Investments		5,479.40	
Accrued Interest and Premium		466.01	
Seaview Golf Club, see contra		279.30	
Refund of Excess Remittances		28.35	
Miscellaneous		173.73	
Reprints published for cooperating societies, at cost		2,150.24	
Total disbursements		$99,303.01	99,303.01
Cash on hand December 31, 1924			$282.06

FINANCIAL CONDITION AT CLOSE OF FISCAL YEARS 1920–1924, INCLUSIVE.

Fiscal Year.	ASSETS.					Liabilities.	Surplus.[b]
	Cash Balance.	Investment.	Accounts Receivable.	Advance Rent and Furniture and Fixtures.	Total.	Accounts Payable.[a]	
1920	$265.05	$17 608.09	$3 823.90	$5 897.48	$27 594.52	$2 608.50	$24 986.02
1921	3 819.07	17 608.09	5 218.35	5 121.34	31 766.85	770.00	30 996.85
1922	3 495.03	20 660.65	5 242.65	4 293.09	33 691.42	663.25	33 028.17
1923	5 139.72	23 660.65	5 202.16	3 598.49	37 601.02	467.99	37 133.03
1924	282.06	19 845.44	6 909.42	3 672.90	30 709.82	4 797.31	25 912.51

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[a] These sums represent dues and publications paid in advance and do not include any unpaid bills except for 1920 loan of $2200 from the Girard Trust Company, and for 1924 a loan of $4000.

[b] These items include $1300 on account of Life Memberships for 1920 to 1923, inclusive, and $1600 for 1924.

the original five-year lease for Society headquarters, which terminated December 31, 1924) and $850 rental for 1924 for the additional floor of the Society headquarters acquired January 1, 1924. Beginning with January 1, 1925, the Society's annual rental for headquarters will be $2400 as explained in the annual report a year ago.

Inventory of Publications in Stock:

In the foregoing financial analysis no account has been taken of the assets of the Society in the form of publications in stock. The inventory on May 15, 1925, may be summarized as follows:

	NUMBER OF COPIES
Proceedings	5178
1924 Book of Standards	2388
Index to Proceedings (Vols. I–XII, incl)	566
(Vols. XIII–XX, incl.)	725
Reports of Committee D-1 (1903–1914, incl.)	276
Reprints of 220 standards (approximate)	13000
Special reprints for general sales (approximate)	7200

In order to provide storage space for current publications and to equalize the stock of back publications, there was distributed during the year to the members about 2900 volumes of Proceedings covering the years 1905 to 1915, inclusive.

Letter Ballot on Adoption of Standards:

The Executive Committee has made the ruling that hereafter the letter ballots taken upon the adoption of proposed new and revised standards, after the annual meeting, shall be signed by each member voting a ballot. This ballot has never been a secret one and it has heretofore been the policy to require the signature of the member on the envelope in which the ballot was enclosed; but as a readier means of identification it has been decided to arrange to have this signature placed on the ballot itself.

Research in the Society:

The consideration of problems relating to the development of research within the Society and the promotion of knowledge of the materials of engineering, as discussed in previous reports of the Executive Committee, is now proceeding upon a satisfactory basis through the activities of Committee E-9 on Correlation of Research, organized at the annual meeting last year with the following personnel

Breyer, F. G.
Burgess, G. K.
Clamer, G. H.
Moore, H. F. (*Chairman*)
Schmitt, F. E.
Warwick, C. L. (*Ex-officio Secretary*)

The first report of the committee, to be presented at this annual meeting, shows that the committee has been considering the problems before it from a broad viewpoint. In particular it has conducted a survey of the research activities now going on in the Society's committees as the necessary first step in the study of existing agencies of the Society for promotion of knowledge of materials and discussion of the question as to what new agencies, if any, are needed in the Society for the development of this work. It is to be remembered that Committee E-9 is not a research committee in the sense of actually conducting research. It is expected to devise ways and means by which the whole question of research activities in the Society's committees and in other channels may be advanced along the soundest lines to the end that the Society will contribute in increasing measure to the knowledge of materials of engineering. The committee will advise the Executive Committee in all matters relating to research and to it will be referred for consideration various problems of this kind.

The Executive Committee has suggested to the officers of the standing committees that they submit annually a statement of problems encountered in the course of their work that suggest the need for investigation, especially where the need arises for information which a standing committee may not be in a position to obtain and which may involve more or less extensive research. Committee E-9 will be in a position to assist standing committees in connection with such problems and to find some means by which proper investigations may be carried out.

One such problem brought to the committee by Committee A-9 on Ferro-Alloys has resulted in the decision to form a Research Committee on the Effect of Tin on High-Speed Tool Steels, which is now in course of organization under the chairmanship of N. B. Hoffman.

The matter of financial support for research, including administrative expenses and the publication of results of research, is receiving consideration in both Committee E-9 and the Executive Committee.

Proposed Establishment of Annual Medal and Annual Lecture:

As a means of stimulating research in materials and of keeping before the members the fact that an important function of the Society is the promotion of knowledge of engineering materials, Committee E-9 on Correlation of Research made two recommendations to the Executive Committee, namely (1) that an annual Medal be established to be awarded for a paper of outstanding merit constituting an original contribution on research in materials, and (2) the establishment of an annual Lecture to be given at the annual meetings by leaders in the field of engineering materials. These recommendations have been approved by the Executive Committee which has felt that the establishment of such a Medal and Lecture will serve not only the purposes suggested by Committee E-9 but at the same time will afford the opportunity of commemorating for all time the names of the first President of the Society, the late Charles B. Dudley (1842–1909), and the first Secretary of the Society, the late Edgar Marburg (1864–1918). Accordingly, the Executive Committee recommends to the Society that the establishment of the proposed Medal and the proposed Lecture be authorized by the Society at this annual meeting along substantially the following lines:

CHARLES B. DUDLEY MEDAL

1. The Medal shall be known as the Charles B. Dudley Medal, taking the name of the first President of the Society. Doctor Dudley, whose most distinguished service was as chemist of the Pennsylvania Railroad from 1875 until his death, served as President of the Society from 1902 until 1909. His inspiring leadership during these early years of the Society has had a profound influence on its development. Himself a noted research worker and investigator, it seems most fitting to the Executive Committee that this Medal should bear his name.

2. The Medal is established as an annual award to the author or authors of a paper, presented before the Society, of outstanding merit constituting an original contribution on research in engineering materials, with the understanding that if no paper in any given year seems to merit this distinction the award will not be made.

3. The Executive Committee shall prepare rules under which the award shall be made and shall appoint annually a Committee on Award of the Charles B. Dudley Medal, this committee to consist of three members.

EDGAR MARBURG LECTURE

1. The Lecture shall be known as the Edgar Marburg Lecture, taking the name of the first Secretary of the Society. Serving as Secretary from 1902 until his death in 1918, Doctor Marburg put the work of the Society on a firm foundation and through his development of the technical programs he brought wide recognition to the Society as a forum for the discussion of properties and tests of engineering materials. His valued services in this direction and his own worthy contributions to the knowledge of materials are fittingly recognized in the proposal to identify his name with this Lecture.

2. The purpose of the Lecture shall be to have described before the Society by leaders in their respective professions outstanding developments in the promotion of knowledge of engineering materials.

3. The Lecture shall carry with it an honorarium of $200 and a suitably engraved certificate.

4. The Executive Committee shall prepare rules under which the Lecture shall be given and shall appoint annually a Committee on the Edgar Marburg Lecture whose duty it shall be to select the lecturer, the committee to consist of three members.

5. In the selection of the lecturer consideration shall be given to the plan of securing lecturers from foreign countries at suitable intervals and as the funds permit.

To establish the Medal and Lecture upon a proper basis will require respectively at least $1500 and $4500, or a minimum total of $6000. The Executive Committee is confident that the objects of the Medal and Lecture and the proposal to commemorate the names of Dudley and Marburg will meet with the hearty approval of the members of the Society and that the members will deem it a privilege to raise the funds required for this purpose. Accordingly the Executive Committee recommends that the Medal and the Lecture be established by voluntary contributions from Society members and that the effort be made to have as many members as possible subscribe to the fund. If this recommendation meets with approval the Executive Committee would propose that it be authorized to appoint a suitable committee to assume charge of the collection of this fund and that the interest and support of the standing committees of the Society be enlisted in this project.

Promoting the Usefulness of the Society's Work:

The Society at the annual meeting in 1924 referred to the Executive Committee for its consideration a paper presented at that meeting by Thomas H. Wiggin on the subject "How Shall the Benefit of A.S.T.M. Standardization be Secured to the Small User." Consideration of the points developed in this paper led the Executive Committee to conclude that it will be desirable to study the broad problem of increasing the usefulness of the Society to its own members and to industry. Discussion of the matter developed a number of suggestions for making our work better known and our standards more widely used, and it was determined to appoint a Special Committee on Promotion of Usefulness to consider this problem in its various aspects and to make recommendations thereon to the Executive Cmmittee. The following committee was appointed:

Chandler, W. L.
Farmer, F. M. (*ex-officio*)
Gibboney, J. H. (*chairman*)
Kinney, W. M.
Lynch, T. D.
Marston, Anson
Rys, C. F. W.
Warwick, C. L. (*ex-officio*)
Wiggin, T. H.

This committee was instructed to study the whole problem of increasing the usefulness of the Society and to it was referred Mr. Wiggin's paper. The committee has held two meetings at which a number of matters were discussed, the principal ones being: Greater publicity of the general activities of the Society, both of the work accomplished at the annual meeting and of the activities of the Society and its committees between annual meetings; a plan of bringing the work of the Society, and especially the value of its specifications and methods of test, to the attention of the faculties and students of technical schools and colleges; the determination in so far as possible of the extent to which the standards of the Society are being used in industry; the desirability of having our members and others make as much use as possible of A.S.T.M. specifications in their own specifications; means by which our work may be made more widely known to the buying and selling public; means by which the small consumer especially may more greatly benefit by the standardization work of the Society than is apparently the case at present; and extending the usefulness of the Society in certain geographical districts, of which the most notable example is that of the Pacific Coast section.

While most of these matters are still under discussion, the Special Committee made certain recommendations during the year to the Executive Committee, which were approved and have been or will be carried out at the appropriate time. These relate to the following:

Publicity.—A procedure is being established in the office of the Secretary-Treasurer for securing accounts from time to time of the activities of standing committees for general publicity purposes, with the thought of preparing news releases to be sent to the editors of the technical and trade press and other suitable agencies for publication. The group meetings of the standing committees afforded a particularly advantageous opportunity along this line and the plans were put into successful operation in connection with the meetings of the metals and non-metals committees in Philadelphia in March. With the cooperation of the officers of the standing committees that met at that time a complete news account of each committee meeting was prepared and mailed the day following the group meetings to the editors of about thirty technical and trade journals. Many of these journals carried a complete account of the meetings. In addition, appropriate accounts of the meetings written from a semi-popular point of view were prepared for the Philadelphia newspapers and resulted in very satisfactory mention of these meetings in the daily press. It is believed that the opportunities afforded for publicity by these group meetings, supplemented by news releases at various times as the activities of the several committees may make it desirable, will for the time being adequately take care of this feature.

Plans for securing wider publicity of Society activities as expressed by the proceedings of the annual meeting are being developed along lines somewhat similar to those mentioned above for the group committee meetings.

The Executive Committee has authorized the reprinting of a paper on "Standard Specifications and Methods of Test for Materials" written by the Secretary-Tresaurer and presented at the Pan-American Standardization Conference in Lima, Peru, December, 1924. The paper discusses the activities of the Society and the general problem of standardization and its value to industry, and the Executive Committee believes that it can be effectively used in furthering the work of the Society and promoting its general usefulness.

Technical Schools.—The Executive Committee has authorized the preparation of a suitable pamphlet describing the work of the Society as the first step in the above-mentioned program of bringing the work of the Society more immediately to the attention of the faculties and students of technical schools. To establish such contacts with technical schools is considered by the Executive Committee to be one of the most fruitful means of bringing about a greater appreciation of the value of standard specifications and tests for materials and of the need for accurate knowledge of the properties of materials, on the part of those who in the years to come will hold the responsible positions in industry. While the plans for this work are not completely formulated, they contemplate having a number of our members give talks to engineering students on various phases of materials engineering in which the important part that the work of the Society plays in this field will be brought out.

In this connection the Special Committee will consider a suggestion made by Committee A-4 on Heat Treatment of Iron and Steel that steps be taken to interest engineering students in Society membership, possibly through a class of Student Membership. The Special Committee is also considering a request from the Society for the Promotion of Engineering Education for assistance in the survey they are conducting of the occupational demands for engineering graduates.

Survey of Extent of Use of A.S.T.M. Standards.—There is a feeling, for which some justification exists, that many of our standards are not used as extensively as their industrial and economic value warrants. To be of any real value, standards must be used, and the value of our standardization work to the members and to industry is increased by doing whatever we can as a society to have our standards used more extensively. In considering means whereby the usefulness of the Society's standards may be promoted, the Special Committee believes it desirable to obtain some indication, however approximate, of the extent of their use at present. It was considered that such a survey may best be made by the standing committees themselves, for they have developed the standards and are directly in touch with the producers and consumers of the commodities involved. Accordingly the standing committees have been asked to ascertain the desired information in whatever way may seem most appropriate in view of the particular conditions surrounding the work of each committee. Such a survey is actually under way in a number of committees and it is expected that information of value may be in hand for study within the next few months.

The importance of the problems being studied by this Special Committee is self-evident and the Executive Committee will welcome suggestions from any of the members relative to these problems.

Increased Financial Support of Society Activities:

The Executive Committee has given some consideration to a problem that it believes must be faced as the Society continues to grow and to develop its work in standardization and the promotion of knowledge of materials, and which is typified by the conditions in 1924 when the volume of technical data of the kind that the Society should publish if it is properly to cover its field considerably exceeded the volume that could be printed from the current funds available for that purpose. The problem is primarily that of securing a greater income for the Society in order that it may properly discharge the functions for which it is created. This problem is being carefully studied and although the Executive Committee is not prepared to make any recommendations at this time it does wish to make a statement of the problem as it sees it for the consideration of the members.

Our efforts for many years have been more or less concentrated, so far as active committee work and actual functioning of the Society are concerned, upon that one of our two main functions designated as standardization of specifications and methods of test for materials. We have published all the reports our committees have brought to us and all of the specifications and methods of test that have been adopted by the Society. Part I of the Proceedings (containing committee reports and new and revised tentative standards), the triennial Book of A.S.T.M. Standards, with its supplements in the intervening years, and the special publication annually of the Book of A.S.T.M. Tentative Standards, are the tangible evidence in the form of publications of that phase of our work. With expansion of the Society's activities, the creation of new committees, and the increasing interest in standardization work that has been shown by many industries, it is to be expected that there will be a continued growth in our standardization activities.

This work involves certain publication problems, as for example the method of publishing the Book of Tentative Standards. These problems will have to be studied with due regard to their financial aspects; but the Executive Committee believes that the normal growth of the Society may fairly be expected to provide the income necessary to meet increased expenses of our standardization activities.

Paralleling this increase in the standardization activities and the publications resulting therefrom, there has been within the past two or three years a noticeable increase in the volume of technical data brought to the Society for publication, in the form principally of the technical papers and discussions but also through reports of investigations by the standing committees that do not relate to their standardization work. The publication and dissemination of such data is an important function of the Society. Last year the amount of such data, as explained earlier in this report in the section on "Publications," was so great that a deficit was incurred. While that condition was to some extent exceptional in that a number of important projects seemed to come to head at the same time, it is to some degree an indication of future developments as the Society grows. It follows necessarily that stimulation of the work of promoting knowledge of materials, such as will be the outcome of the activities of Committee E-9 on Correlation of Research, will result in there being brought to the Society for publication and dissemination more and more data on the properties of materials. Also it is desirable, as pointed out in the report of the Executive Committee a year ago, that the standing committees should include in their reports the principal data upon which proposed specifications and methods of test or revisions thereof have been based; and while such data if extensive and in considerable detail need not be published *in extenso*, much of it has permanent value and should be made available. The particular situation brought about by the greatly increased volume of the Proceedings for last year has been met by a corresponding curtailment of the volume of Proceedings for this year. This has meant the rejection of a number of papers offered for publication, most of them of considerable promise; and such curtailment of committee reports as has seemed possible. The

Executive Committee believes that it is contrary to the best interests of the Society to have to decline the publication of such data on the plea that it has not the necessary funds.

While the problem as presented in the preceding paragraphs has been discussed from the point of view of necessity for additional income, the Executive Committee, realizing that the problem has two sides,—that is, increasing the income or decreasing the expenses of publication,—is also studying the publication policies of the Society in the light of some recent representations of the Committee on Papers and Publications with particular reference to the publication and distribution of the standards and tentative standards.

American Engineering Standards Committee:

Past-President J. A. Capp has been re-appointed as the Society's representative on the Standards Committee for a term of three years. Past-Presidents A. A. Stevenson and C. D. Young are the other representatives on this Committee. Mr. Capp is the Society's representative on the A.E.S.C. Executive Committee.

At the present time 39 A.S.T.M. standards have been approved by the Standards Committee, one as American Standard and 38 as Tentative American Standard, and three are still under consideration. The Society has been appointed as sponsor for 30 of these and joint sponsor for three. Sponsorships for the remaining six are still under consideration. No A.S.T.M. specifications have been submitted to the Standards Committee since the last annual meeting pending a study of the methods by which the A.E.S.C. sectional committee procedure can be coordinated with the Society's standardization work as at present conducted without interfering to any serious degree with the present efficient and effective work of our committees.

Sectional Committees for which the Society is now sponsor or joint sponsor are those on Zinc and Zinc Ores, Insulated Wires and Cables for Other than Telephone and Telegraph Use, Methods of Testing Wood, Standards for Drain Tile, Numbering of Steels and Fire Tests of Materials and Construction. Reports of such meetings of these committees as have been held during the year have been received and considered by the Executive Committee. The Sectional Committee on Drain Tile is conducting a ballot upon the submission of the revised Drain Tile Specifications, adopted by the Society last year, for submission to the Engineering Standards Committee for approval as American Standard. The Sectional Committee on Methods of Testing Wood, which has brought out through the Society Tentative Methods of Testing Small Clear Specimens of Timber

(D 143 – 24 T) and Tentative Methods of Conducting Static Tests of Timber in Structural Sizes (D 198 – 24 T), is not yet prepared to recommend their advancement to standard, and a report of progress has been submitted to the Standards Committee.

The Sectional Committee on Numbering of Steels has held one meeting during the year, as a result of which an investigation is being made of the number of existing specifications for steels under the scope of the Sectional Committee with the thought that if the number of such specifications could be materially reduced the work of the Sectional Committee in developing a system of numbering such steels would be much facilitated, as it is required that any numbering system be devised upon definite specifications.

The Sectional Committee on Fire Tests of Materials and Construction has held three meetings during the year at which substantial progress has been made in the study of the Tentative American Standard Specifications for Fire Tests of Materials and Construction (A.S.T.M. Serial Designation: C 19 – 18).

During the year the Society has been engaged in the preliminary steps involved in the organization of the Sectional Committee on Zinc Coating of Iron and Steel, for which it is the sponsor. The personnel of the committee after some delays has been practically completed and the meeting for organization is planned in the near future.

Since the last annual meeting, the Society has accepted joint sponsorship with the American Gas Association, New England Water Works Association and American Water Works Association for the organization of a Sectional Committee on Cast-Iron Pipe. Invitations have been extended to a number of organizations to become members of this committee and the meeting for organization will be held as soon as possible.

The activities of the Sectional Committe on Aeronautical Safety Code, upon which the Society is represented by the Secretary-Treasurer, have led to the preparation of an Aeronautical Safety Code, which after approval by the sponsors (the U. S. Bureau of Standards and the Society of Automotive Engineers) will be submitted to the Standards Committee for approval. This Code deals only in a very general way with specifications and tests for materials, and the specifications of the Society are mentioned as recognized specifications issued by a responsible organization and giving determinate properties of materials.

With respect to the Sectional Committee on Shafting, upon which the Society is represented by F. M. Waring and J. J. Shuman, Committee A-1 on Steel is studying a re-arrangement of certain of its specifications (for screw stock and commercial bar steel) so as to include in a logical way specification requirements for shafting stock. Similarly, in connection with the activities of the Sectional Committee on Pipe Flanges and Fittings, upon which the Society is represented by R. A. Bull and V. T. Malcolm, Committee A-1 is preparing for submission as tentative this year Specifications for Alloy-Steel Bolting Material for High-Temperature Service and Specifications for Carbon-Steel Castings for Valves, Flanges and Fittings for High-Temperature Service. The Sectional Committee on Specifications for Special Trackwork Material, on which the Society is represented by E. F. Kenney, has been particularly active during the year in the preparation of various specifications, in which so far as possible existing A.S.T.M. standards have been used either *in toto* or as a basis for the special material specifications with which this committee is working.

During the year the following appointments were made of representatives of the Society on the following sectional committees under the sponsorship of other organizations: E. S. Taylerson, succeeding D. M. Buck, deceased, on the Sectional Committee on Zinc and Zinc Ores, under the joint sponsorship of the A.S.T.M. and the American Zinc Institute; R. A. Bull, succeeding H. V. Wille, on the Sectional Committee on Pipe Flanges and Fittings, under the joint sponsorship of the American Society of Mechanical Engineers, Committee of Manufacturers on Standardization of Fittings and Valves, and Heating and Piping Contractors National Association; F. N. Speller on the Sectional Committee on Tubular Steel Poles, under the sponsorship of the American Electric Railway Association; S. S. Sunderland on the Sectional Committee on Wire Rope for Mines, under the sponsorship of the American Mining Congress; and R. E. Hess on the Sectional Committee on Engineering Symbols and Abbreviations, under the joint sponsorship of the American Association for the Advancement of Science, the American Society of Mechanical Engineers, the American Society of Civil Engineers, the American Institute of Electrical Engineers and the Society for Promotion of Engineering Education.

Pan-American Conference on Standardization:

Through the invitation of the American Engineering Standards Committee the Society was represented in the development of the program of the Pan-American Conference on Standardization held in Lima, Peru, December, 1924, under the auspices of the Pan-American Union and the Inter-American High Commission. Upon invitation of the Director General of the Pan-American Union, the Secretary-

Treasurer prepared a paper on "Standard Specifications and Methods of Test for Materials," which was presented at the Conference. A report of the proceedings of the Conference has been received and is under consideration by the Executive Committee at this time.

Directory of Specifications:

The Society has continued its cooperation with the U. S. Department of Commerce in the preparation of a Directory of Specifications for supplies purchased by Federal, state and municipal governments and public institutions, with the object in view of bringing about the wider use of specifications by these agencies as a basis of purchase for supplies. Excellent progress has been made during the year in the preparation of this Directory, in which will be listed and suitably classified all the commodity specifications in general use in the United States. It is expected the Directory will be available the latter part of this year. Vice-President W. H. Fulweiler represents the Society on the advisory committee that is cooperating with the Department of Commerce in this work. Mr. Fulweiler as a member of this committee has recommended that there be prepared to go with the Directory a Treatise on Specifications that would cover such matters as the general form and development, interpretation, use, limitations and choice of specifications, as a guide to the users of the Directory. The Executive Committee has offered the assistance of the Society to the Department of Commerce in the preparation of such a Treatise, of which an outline has been prepared and submitted to the Advisory Committee at its request.

Division of Simplified Practice, U. S. Department of Commerce:

The Society has continued contact with the Division of Simplified Practice in connection with those projects in which we are interested. Subjects that have come up during the year in this category are steel reinforcement bars, paving brick, roofing materials and lumber standards, the latter being handled through the Central Committee on Lumber Standards. The committees of the Society dealing with these subjects are expected to report upon them at this meeting. Committee C-2 on Reinforced Concrete formally approved the program of reduction of the number of standard sizes of concrete reinforcement bars, and the Division of Simplified Practice has been so advised.

T. R. Lawson has been appointed as representative of the Society on the Permanent Committee on Standardization of Paving Brick Varieties, to succeed W. P. Blair, deceased.

Division of Engineering, National Research Council:

Through its representatives the Society has continued to maintain contacts with the Division of Engineering and its various committees, especially the Advisory Board on Highway Research, the Bureau of Welding and the Committee on Corrosion. Alexander Milne has been appointed to succeed H. L. Whittemore as our representative on the Committee on Welding Wire Specifications of the Bureau of Welding.

International Annual Tables of Constants and Numerical Data:

The Society is supporting the publication of the International Annual Tables of Constants and Numerical Data through an annual contribution of $100 to the American Advisory Committee appointed by the National Research Council. These Tables of physical, chemical and technological data are published by an International Commission under the authority of the National Research Council and the International Union of Pure and Applied Chemistry. Vol. V, containing data for 1917–1922, has been printed and a notice of this volume with an order blank was included in each copy of the 1924 Proceedings of the Society. Under the terms of a reciprocal agreement, a notice of the Society's publications appears in the volume of Annual Tables.

Vols. VI and VII of the Tables, containing respectively the data for 1923 and 1924, are in process of compilation.

U. S. National Committee of the International Electrotechnical Commission:

The Society has been able to be of considerable assistance to the U. S. National Committee of the International Electrotechnical Commission, upon which it is represented by the Secretary-Treasurer, in the preparation of a report on the testing of transformer and other insulating oils for the use of the committee at the meeting of the International Commission held at The Hague in April. The entire subject was reviewed by a special committee appointed by the President under the chairmanship of C. E. Skinner, and a comprehensive report on the subject based upon the work of Committee D-9 on Electrical Insulating Materials was adopted by the U. S. National Committee and presented at the International Conference. E. A. Snyder, a member of Committee D-9, attended The Hague conference as an official delegate and representing the Society.

Miscellaneous Joint Committees:

The Society has continued, through its representation, participation in the work of the various joint committees upon which it is represented. The Joint Committee on Concrete Culvert Pipe, the Joint Committee on Investigation of the Effect of Phosphorus and Sulfur in Steel and the Joint Research Committee on the Effect of Temperature upon the Properties of Metals have been active during the year. Joint committees with the American Electric Railway Engineering Association on trolley wire and on girder rails have been active, the work of the former having resulted in complete agreement between the two societies on specifications for copper trolley wire and substantial progress towards agreement on specifications for bronze trolley wire. A few differences that still exist between the specifications of the two bodies for girder rails are being considered by the latter committee.

The Executive Committee has recently authorized the formation of a joint committee with the American Wood Preservers' Association to consider the development of methods of test for petroleum oils for wood preservation, upon which the Society's representation will be through members appointed from Committees D-2 on Petroleum Products and Lubricants and D-7 on Timber.

Other Cooperative Work:

Among other cooperative work that has taken place this year should be mentioned the following: The Secretary-Treasurer has been appointed to represent the Society on the Executive Board of the American Marine Standards Committee, which is considering marine standardization that will include some specifications for materials. M. E. McDonnell has been appointed to represent the Society on a Research Committee on Metal Springs of the American Society of Mechanical Engineers. Similarly, James Aston has been appointed to represent the Society on the Corrosion Committee of the American Society of Refrigerating Engineers. Don L. Quinn has been appointed to represent the Society on the committee of the Transportation and Domestic Commerce Division of the Bureau of Foreign and Domestic Commerce that is to make a study of the methods of packing goods for domestic shipment.

Translation of Standards:

The translation by the Bureau of Foreign and Domestic Commerce of about thirty of the Society's standard specifications into Portuguese for use principally in connection with export business in Brazil has been completed and the translated specifications are now being printed. The first two of this series, Specifications for Carbon-Steel Rails and Specifications for Low-Carbon-Steel Splice Bars, have recently been received. The specifications are printed in both English and Portuguese with an appropriate prefatory note explaining the purpose of the series.

Revision of a number of the more important specifications translated into Spanish some years ago is now being undertaken so that the Spanish-English editions of these specifications issued by the Bureau of Foreign and Domestic Commerce will be brought up to date with the 1924 Book of Standards.

International Association for Testing Materials Formally Dissolved:

The Executive Committee has received a formal communication from Ernst Reitler, General Secretary of the former International Association for Testing Materials, advising that final steps had been taken for the formal dissolution of the International Association. The identification in the list of members in the Year Book of members who held membership in the International Association will now be discontinued.

Respectfully submitted on behalf of the Executive Committee,

F. M. FARMER,
President.

C. L. WARWICK,
Secretary-Treasurer.

REPORT OF COMMITTEE A-1
ON
STEEL

During the past year Committee A-1 has held two meetings, one on October 24, 1924, and the second on March 20, 1925. A third meeting will be held prior to the presentation of this report to the Society, at which the report will be reviewed and other matters considered.

The Advisory Committee has held three meetings, at which matters concerning the general administrative affairs of the committee were considered.

The present membership of the committee is 171, of which 89 are classed as non-producers and 82 as producers.

RECOMMENDATIONS AFFECTING STANDARDS AND TENTATIVE STANDARDS

The recommendations of the committee affecting standards and tentative standards are presented first in summarized form together with an analysis of the letter ballot on each item. They are then referred to, where necessary, in greater detail, being grouped for convenience in the order of the respective sub-committees directly responsible for them.

I. Proposed Revisions in Standards.—The committee recommends that the revisions in the following standards, which are given in full in the Appendix to this report, be published as tentative for one year before referring them to letter ballot of the Society for adoption as standard:

1. For Carbon-Steel and Alloy-Steel Forgings (A 18 – 21).
2. For Quenched-and-Tempered Carbon-Steel Axles, Shafts and Other Forgings for Locomotives and Cars (A 19 – 21).
3. For Carbon-Steel Forgings for Locomotives (A 20 – 21).
4. For Quenched-and-Tempered Alloy-Steel Axles, Shafts and Other Forgings for Locomotives and Cars (A 63 – 21).
5. For Carbon Steel Car and Tender Axles (A 21 – 18).
6. For Welded and Seamless Steel Pipe (A 53 – 24).
7. For Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 24).

II. Proposed New Tentative Standards.—The committee recommends that the following new specifications, which are appended to this report,[1] be accepted for publication as tentative:

8. For Structural Silicon Steel.
9. For Carbon-Steel Castings for Valves, Flanges and Fittings for High-Temperature Service.
10. For Alloy-Steel Bolting Material for High-Temperature Service.

III. Tentative Standards.—The committee recommends that the following tentative standards remain as tentative for another year:

For Steel Tie Plates (A 67 – 20 T).
For Carbon Tool Steel (A 71 – 24 T).
For High-Speed Tool Steel (A 92 – 24 T).
For Low-Carbon-Steel Track Bolts (A 76 – 20 T).
For Electric Cast-Steel Anchor Chain (A 77 – 20 T).
For Steel Plates of Structural Quality for Forge Welding (A 78 – 23 T).
For Steel Plates of Flange Quality for Forge Welding (A 89 – 23 T).
For Cold-Drawn Steel Wire for Concrete Reinforcement (A 82 – 21 T).
For Carbon-Steel Castings for Railroads (A 87 – 22 T).

The above recommendations have been referred to letter ballot of the committee, which consists of 171 members: 146 ballots have been cast, 25 members having refrained from voting. The analysis of the vote of the committee is given in the following table:

TABLE I.—ANALYSIS OF VOTE ON REPORT OF COMMITTEE A-1.

Items	Affirmative	Negative	Not Voting[a]
I. PROPOSED REVISIONS IN STANDARDS			
1. For Carbon-Steel and Alloy-Steel Forgings (A 18 – 21)	94	1	51
2. For Quenched-and-Tempered Carbon-Steel Axles, Shafts and Other Forgings for Locomotives and Cars (A 19 – 21)	89	1	56
3. For Carbon-Steel Forgings for Locomotives (A 20 – 21)	88	1	57
4. For Quenched-and-Tempered Alloy-Steel Axles, Shafts and Other Forgings for Locomotives and Cars (A 63 – 21)	89	2	55
5. For Carbon-Steel Car and Tender Axles (A 21 – 18)	88	0	58
6. For Welded and Seamless Steel Pipe (A 53 – 24)	85	1	60
7. For Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 24)	89	1	56
II. PROPOSED NEW TENTATIVE STANDARDS			
8. For Structural Silicon Steel	86	1	59
9. For Carbon-Steel Castings for Valves, Flanges and Fittings for High-Temperature Service	80	3	63
10. For Alloy-Steel Bolting Material for High-Temperature Service	76	2	68

[a] In this column are recorded the number of votes cast as "Not Voting" on the various items.

Total number of ballots cast	146
Number of members who failed to vote	25
Total membership	171

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[1] See pp. 514–527.—ED.

The committee passed a resolution to the effect that Sub-Committee X on Automobile Steels be discontinued and that the Standard Specifications for Automotive Carbon and Alloy Steels (A 29 – 24) be withdrawn from the Book of Standards at the next printing. This recommendation is made because, although these specifications originated in Committee A-1, the subsequent activity of the Society of Automotive Engineers in the drafting of specifications has brought about a situation wherein all ordering of automotive steels is to S.A.E. specifications, thus making the Society's specifications really unnecessary. The recommendation is made with the understanding that it is not intended to be a precedent for the withdrawal on the part of Committee A-1 from other activities relating to specifications and methods of test for materials in the automotive field.

In the following the committee presents the foregoing recommendations in detail where necessary, as well as other features of its work, grouped for convenience in the order of the respective sub-committees responsible for them.

SUB-COMMITTEE II ON STRUCTURAL STEEL FOR BRIDGES, BUILDINGS AND ROLLING STOCK

This sub-committee has submitted new Tentative Specifications for Structural Silicon Steel appended hereto.[1] This type of steel has been used on various important bridges and is being used on the new Delaware River Bridge. The physical properties are higher than those of ordinary carbon steel, structural grade, and there is apparently a wide field for the use of steel of this type. In the opinion of the engineers who are familiar with its use, the committee is warranted in submitting these specifications to the Society so that they may be available for use, or for discussion and improvement as experience with the material develops.

SUB-COMMITTEE VI ON STEEL FORGINGS AND BILLETS

Sub-Committee VI is recommending a revision in the section covering prolongations for tests called for in its forging specifications, and has also been actively engaged in the revision of the Standard Specifications for Carbon-Steel Car and Tender Axles (A 21 – 18) to bring them into agreement with the specifications of the American Railway Association. A complete revision of these specifications is submitted herewith.[2]

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[1] See p. 514.—ED.

[2] See p. 509.—ED.

SUB-COMMITTEE IX ON STEEL TUBING AND PIPE

This sub-committee is recommending a change in the scope of the Standard Specifications for Welded and Seamless Steel Pipe (A 53 – 24) and is recommending the insertion of a new paragraph under Section 16 covering the gaging tolerances in the Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 24).

SUB-COMMITTEE XXII ON PIPE FLANGES AND FITTINGS

This sub-committee has effected a satisfactory organization and has done considerable work during the past year in the development of specifications for suitable materials for valves, flanges, fittings and pipe for high-temperature service in both central stations and oil refineries. The use of high temperatures and pressures in power plants is developing very rapidly and the conditions require the development of specifications to cover materials that are to be subject to such severe service. This subject is perhaps the most important one before Committee A-1 at this time. The sub-committee has submitted proposed Tentative Specifications for Carbon-Steel Castings for Valves, Flanges and Fittings for High-Temperature Service up to and including 750° F., and proposed Tentative Specifications for Alloy-Steel Bolting Material for the same service. These specifications are appended to this report.[1] In presenting these proposed tentative standards to the Society, the committee wishes to point out that the tentative standards are not considered as being the final word on the subject, but they are the result of considerable work and discussion in the sub-committee and represent the best specifications that the committee has been able to prepare up to the present time. It is desired that they be accepted by the Society so that they may be printed and be available for the information of all who are interested in this subject, to the end that discussion may be stimulated and constructive criticism developed that will be of benefit to the sub-committee in their further consideration of the subject.

This report has been submitted to letter ballot of the committee, which consists of 171 members, of whom 110 have voted affirmatively, none negatively, and 61 have refrained from voting.

Respectfully submitted on behalf of the committee,

F. M. WARING,
Chairman.

G. H. WOODROFFE,
Secretary.

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[1] See pp. 519–527.—ED.

EDITORIAL NOTE

The proposed revisions of the standard specifications listed in this report as items 1 to 4, and 6 to 7, page 70, were accepted for publication as tentative and appear on pages 895 to 896. The proposed revisions of the Standard Specifications for Carbon-Steel Car and Tender Axles were accepted for publication as new tentative specifications and appear on page 509.

The proposed Tentative Specifications for Structural Silicon Steel; for Carbon-Steel Castings for Valves, Flanges and Fittings for High-Temperature Service; and for Alloy-Steel Bolting Material for High-Temperature Service were accepted for publication as tentative and appear on pages 514, 519 and 523, respectively.

APPENDIX

PROPOSED REVISIONS IN STANDARDS FOR STEEL

In this appendix are given proposed revisions in certain standard specifications, which, for convenience, are numbered from 1 to 7, inclusive, to correspond to the report of the committee. In connection with each title is given the reference to the publication in which the specifications appear in their present form.

No. 1.—STANDARD SPECIFICATIONS FOR CARBON-STEEL AND ALLOY-STEEL FORGINGS: A 18 – 21[1]

No. 2.—STANDARD SPECIFICATIONS FOR QUENCHED-AND-TEMPERED CARBON-STEEL AXLES, SHAFTS, AND OTHER FORGINGS FOR LOCOMOTIVES AND CARS: A 19 – 21[1]

No. 3.—STANDARD SPECIFICATIONS FOR CARBON-STEEL FORGINGS FOR LOCOMOTIVES: A 20 – 21[1]

No. 4.—STANDARD SPECIFICATIONS FOR QUENCHED-AND-TEMPERED ALLOY-STEEL AXLES, SHAFTS, AND OTHER FORGINGS FOR LOCOMOTIVES AND CARS: A 63 – 21[1]

In Specifications A 18 – 21 change Section 4, and in Specifications A 20 – 21 change Section 5 from their present form: namely,

> "The manufacturer and the purchaser shall agree upon forgings on which a prolongation for test purposes shall be provided."

and in Specifications A 19 – 21 and A 63 – 21 change Section 4 from its present form: namely,

> "For test purposes, a prolongation shall be left on each forging, unless otherwise specified by the purchaser."

to read as follows:

> "Unless otherwise specified, for test purposes at least 20 per cent of the forgings shall be provided with prolongations or, at the manufacturer's option, a forging may be selected."

In Specifications A 18 – 21 and A 20 – 21 change Section 17 (*a*), and in Specifications A 19 – 21 and A 63 – 21 change Section 19 (*a*) from their present form: namely,

> "The inspector representing the purchaser shall have free entry at all times while work on the contract of the purchaser is being performed, to all

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[1] 1924 Book of A.S.T.M. Standards.

parts of the manufacturer's works which concern the manufacture of the forgings ordered. The manufacturer shall afford the inspector, free of cost, all reasonable facilities to satisfy him that the forgings are being furnished in accordance with these specifications. Tests and inspection at the place of manufacture shall be made prior to shipment."

to read as follows:

"The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the forgings ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the forgings are being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works."

NO. 5.—STANDARD SPECIFICATIONS FOR CARBON-STEEL CAR AND TENDER AXLES: A 21 - 18[1]

These specifications have been extensively revised and are appended hereto[2] in their proposed revised form.

NO. 6.—STANDARD SPECIFICATIONS FOR WELDED AND SEAMLESS STEEL PIPE: A 53 - 24[1]

Section 1.—Change from its present form: namely,

"These specifications cover 'standard,' 'extra strong,' and 'double extra strong' welded and seamless steel pipe. Pipe ordered under these specifications is intended for bending, flanging and other special purposes."

to read as follows:

"*Scope.*—These specifications cover 'standard weight,' 'extra strong,' and 'double extra strong' welded and seamless steel pipe. Pipe ordered under these specifications is intended for coiling, bending, flanging and other special purposes. Butt-welded pipe is not intended for flanging."

NO. 7.—STANDARD SPECIFICATIONS FOR LAP-WELDED AND SEAMLESS STEEL AND LAP-WELDED IRON BOILER TUBES: A 83 - 24[1]

Section 16.—Insert a new Paragraph (*e*) to read as follows, relettering the subsequent paragraph accordingly:

"(*e*) The gaging tolerances in Paragraphs (*b*), (*c*) and (*d*) apply only to tubes as rolled or drawn and before swaging and expanding."

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[1] 1924 Book of A.S.T.M. Standards.

[2] See p. 509.—ED.

REPORT OF COMMITTEE A-2
ON
WROUGHT IRON

During the year, Committee A-2 has held two meetings: one at Washington, D. C., and one at Philadelphia, Pa. Both meetings were well attended and much interest developed for a through investigation of the properties of wrought iron, looking to a more thorough knowledge of wrought iron and the improvement of its physical properties. It is realized that in many respects wrought iron is depending upon its past reputation and the demand for specific data regarding its adaptability to particular fields of service has not been met. Also, there has not been that intensive study of composition and treatment which may better fit it for generally established uses.

It is believed that by the proper annealing treatments of iron, and especially of bolts or staybolts after they have been threaded, a very much longer life will be produced. There is a fertile field for study along this line, and the cooperation of committee members is being enlisted in experimental work on heat treatment and other general research knowledge. This may very materially extend the usefulness of wrought iron and more especially so where shock and vibrations or repeated stresses are the factors, as the final test on any material is its performance in service. Sub-Committee X of Committee A-2 has accordingly been organized for Research Purposes, consisting of both manufacturers and consumers.

SUB-COMMITTEE I ON TUBES AND PIPE

In cooperation with Committee A-1 on Steel the following revisions are recommended:

Standard Specifications for Welded Wrought-Iron Pipe (A 72 - 24).[1]—The following revisions are recommended for publication as tentative:

Section 1.—Change from its present form: namely,

> "These specifications cover 'standard,' 'extra strong' and 'double extra strong' welded wrought-iron pipe."

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[1] **1924 Book of A.S.T.M. Standards.**

to read as follows:

> "These specifications cover 'standard weight,' 'extra strong' and 'double extra strong' welded wrought-iron pipe. Pipe ordered to these specifications is intended for coiling, bending, flanging, and other special purposes. Butt-welded pipe is not intended for flanging."

Section 3 (b).—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> "(b) All pipe [3 in. or under] *under 2 in.* in nominal diameter may be butt-welded, unless otherwise specified. All pipe [over 3 in.] *2 in. or over* in nominal diameter shall be lap welded."

Section 9.—Add a new Section 9 to follow the present Section 8 to read as follows, renumbering the subsequent sections accordingly:

> "In case of doubt as to the presence of steel, the purchaser may, at his own expense, make micrographic examination and chemical analysis to assist in determining whether the material meets the requirements of Section 3 (*a*)."

Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 - 24).[1]—The following revision is recommended for publication as tentative:

Section 16.—Insert a new paragraph (*e*) to read as follows, re-lettering the subsequent paragraph accordingly:

> "(*e*) The gaging tolerances in Paragraphs (*b*), (*c*) and (*d*) apply only to tubes as rolled or drawn and before swaging and expanding."

SUB-COMMITTEE III ON STAYBOLT AND ENGINE-BOLT IRON

Revisions are recommended in the following specifications:

Tentative Specifications for Hollow Staybolt Iron (A 86 - 21 T).[2]—It is recommended that the specifications be revised as follows and continued as tentative in their proposed revised form:

Section 5.—Add a new Paragraph (*c*) to read as follows:

> "In calculating the tensile strength and elastic limit, the area of a $\frac{7}{32}$-in. hole shall be deducted from the area of the bar, but the area of the hole shall be disregarded in calculating the reduction of area."

Section 11.—Change from its present form: namely,

> "The bars shall be truly round within 0.01 in. and shall not vary more than 0.01 in. above, nor more than 0.005 in. below the specified size."

to read as follows:

> "The bars shall be truly round within 0.01 in. and shall be not less than 0.005 in. over, nor more than 0.020 in. over the specified diameter."

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[1] 1924 Book of A.S.T.M. Standards.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 478 (1921); 1924 Book of A.S.T.M. Tentative Standards, p. 49.

Standard Specifications for Staybolt, Engine-Bolt and Extra-Refined Wrought Iron Bars (A 84 - 24).[1]—The following revisions are recommended for publication as tentative:

Section 7.—Add two new Paragraphs (*b*) and (*c*) to read as follows, re-lettering the present Paragraph (*b*) to Paragraph (*d*):

"(*b*) *Grade A.*—For bars 1¼ sq. in. in sectional area, the following deductions from the minimum requirements specified in Section 6 (*a*) shall be made for each square inch of nominal section above 1¼ sq. in.:

Tensile Strength, 250 lb., but not under 46,000 lb.

Reduction of area, 3 per cent, but not under 40 per cent.

"(*c*) *Grade A.*—For flats of all sizes, the minimum reduction of area shall be 40 per cent."

SUB-COMMITTEE V ON CHAIN IRON AND IRON CHAIN

Investigation is now under way for reducing the number of classes of chain covered by the present specifications, also a modification of the proof test to avoid straining in proofing.

An investigation is also being made as to the best method for the annealing of all chains before they are offered for inspection.

SUB-COMMITTEE IX ON METHODS OF CHEMICAL ANALYSIS

Sub-Committee IX on Analysis is working on the determination of the composition of slag and iron and the distribution of the several elements. There is little knowledge regarding the influence of total carbon content of the pig iron upon the physical and chemical properties of the finished wrought iron. Also the varying effect of phosphorus content and its distribution is an important problem in its bearing on the welding qualities and other physical characteristics as well as corrosion resistance of the wrought iron. The causes and preventions of critical red shortness and brittleness are of extreme interest, and important to the manufacturers of certain classes of wrought-iron products. A series of analyses were sent out by Mr. Lundell, the chairman of the sub-committee, to various laboratories. The results showed a fairly uniform check on the various methods of analysis. The following methods of sampling and chemical analysis of wrought iron are presented as information.

PROPOSED METHODS OF SAMPLING AND CHEMICAL ANALYSIS OF WROUGHT IRON

SAMPLING

Remove scale and oil until clean metal is exposed and secure cuttings or drillings that represent the entire cross-section of the specimen. As the various sized particles of wrought-iron cutting or drillings may differ in composition, suitable precautions must be taken to secure all of the cuttings or drillings. For routine work, samples for carbon, manganese, phosphorus, sulfur, and silicon can be taken from the well-mixed drillings. For the determination of slag and oxides or for umpire analyses for the other elements, the entire sample should be weighed and then sifted on a No. 35 sieve (U. S. Standard Sieve Series, nominal openings 0.0197 in.). Each portion so obtained should be thoroughly mixed and weighed. In weighing samples for analysis, portions of these two fractions should be taken in the same proportion which they bear to the gross sample. Thus, if the gross sample, weighing, say 100 g., separates into two portions weighing 80 g. and 20 g. the amount of each that must be weighed to yield a 2-g. portion for analysis are 1.6 g. and 0.4 g., respectively.

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[1] 1924 Book of A.S.T.M. Standards.

METHODS OF CHEMICAL ANALYSIS

The Standard Methods of Chemical Analysis of Plain Carbon Steel (Serial Designation: A 33) of the American Society for Testing Materials[1] may be used for the analysis of wrought iron with the following considerations:

1. For carbon a 3 to 4-g. sample should be used and a blank run made and deducted.
2. For phosphorus a 1-g. sample is satisfactory for the alkalimetric method.
3. For manganese by the Bismuthate Method a 2-g. sample should be used.

DETERMINATION OF SLAG AND OXIDES BY THE IODINE METHOD

REAGENTS REQUIRED

Potassium Hydroxide Solution (10-per-cent).—Make up daily as needed by dissolving 25 g. of KOH in 250 cc. of distilled water.

Iodine.—The iodine used in this method should be resublimed or a blank run made for insoluble material. The purity of different lots of iodine can be checked, conveniently, by heating to volatilize the iodine, dissolving the residue in water, filtering and igniting the insoluble material. The blank on good quality iodine will average about 0.0020 g. on a 22-g. charge.

METHOD

Weigh 4 g. of the drillings as directed under sampling, transfer to a 100-cc. beaker and add 22 g. of resublimed iodine. Add 30 cc. of distilled water to the mixture in the beaker and when the first violent reaction has subsided, wash down the sides of the beaker with a small quantity of distilled water. Place the beaker under a stirring machine and stir vigorously for 1 or 2 hours. When solution is complete, rinse the stirrer and filter the solution by gentle suction through a strong filter paper (such as a Whatman No. 42). Wash the filter and residue with boiling hot distilled water until no trace of iodine remains on the paper and no test for iron is obtained in the washings. Disconnect the filter flask from the source of the suction and fill the funnel to the top of the filter paper with a boiling hot 10-per cent potassium hydroxide solution (see Note 3). Allow the caustic to run through the filter paper by gravity and then repeat the treatment with caustic once more in the same manner. When all of the potassium hydroxide has run through the filter, connect the filter flask to the suction again and wash the filter paper with boiling hot distilled water until the washings do not react alkaline to phenolphthalein. Ignite and weigh as slag and oxides. The weight of the residue divided by 4 and multiplied by 100 gives the percentage of slag and oxides in the sample. The ignited residue can be used for the determination of silica and manganese oxide in the slag.

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[1] 1924 Book of A.S.T.M. Standards.

NOTES

1. Determinations of slag by the above method, or by any of the published methods, are not strictly accurate because:

First.—The iodine treatment leaves other substances than slag (such as base metal silicon and phosphorus) in the insoluble matter;

Second.—The potassium hydroxide treatment not only removes base metal silicon but also more or less of the phosphate, and possibly also silica, in the slag;

Third.—The ignition of the slag results in more or less complete oxidation of any iron occurring as ferrous oxide in the slag. The accuracy of the results can, therefore, not be considered as greater than ±0.3.

2. The practice of adding all of the iodine at one time and of washing with hot water materially shortens the time required for solution and washing and does not introduce any appreciable inaccuracies as compared with the method of cold solution by gradual addition of iodine and cold washing.

3. The caustic treatment is employed to remove any base metal silicon which may have been precipitated during the iodine treatment. Tests have indicated that this treatment also removes varying amounts of P_2O_5 originally present in the slag. This error increases with the phosphorus content of the wrought iron. For high-phosphorus irons it is preferable to treat sufficiently with KOH to leach out practically all of the phosphorus and then to determine the phosphorus present as phosphate in the wrought iron by dissolving the wrought iron in concentrated HCl, boiling to remove phosphine, removing the HCl by several evaporations with HNO_3 and then determining the phosphate in this nitrate solution. The amount of P_2O_5 found should then be added to the ignited residue obtained as described in the method.

4. Variations between different analyses have been due in a large part to the use of dilute acid washes as previously given in certain methods. Tests have shown that even the very dilute mineral acids have an appreciable solvent action on the slag. It is therefore advisable to dispense with any acid washes whatever and use hot distilled water.

SUB-COMMITTEE X ON RESEARCH

This sub-committee is studying the manufacture of wrought iron from various kinds and grades of pig iron. There is also an investigation as to the properties of iron, as to its crystallinity in service as induced by phosphorus or other elements, or by the methods used in manufacture, such as rolling temperatures and cold working.

The committee has investigated the effects of annealing all iron and especially staybolt iron and equalizers as used under railroad equipment. Equalizers which have been thoroughly and carefully annealed are being observed in service and to date no failures have occurred on such annealed equalizers.

Staybolt iron has been investigated both in tension and in vibrating tests which have been conducted both on plain bars and threaded bars. It has been found that little improvement, if any, is made by annealing the plain bar before threading. It is noted, however, that there is an improvement in regard to uniformity where the plain bars are annealed before testing; that the vibrating tests run very much more uniform. It is also found that where staybolt iron is threaded that the vibrating test drops from about 30,000 to around 2500; that the threaded bars have a somewhat higher tensile strength and elastic limit, but the reduction of area and elongation is very materially less. Threaded bars which have been annealed at 1450° F. show that the results in the vibrating test are nearly doubled, that the elongation and reduction of area are very materially increased. The elastic limit and tensile strength are practically the same as before the bar was threaded.

On the Lackawanna Railroad all staybolts placed in service are now being annealed, and it is believed that the life of the staybolt will be considerably increased thereby. It is also found that the bolts are easier to tighten up or peen over in the boiler than bolts not so treated.

The following are the results of some tests on staybolt iron conducted at the Lackawanna Railroad Laboratory. The vibratory tests were made on an Olson testing machine having a $\frac{3}{16}$-in. vibration, under a load of 4000 lb. The length of specimen was 8 in.

Kind of Material	Vibrations to Failure	Elastic Limit, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation, per cent	Reduction of Area, per cent
Plain 1-in. Round	44 341	29 300	47 650	30.50	53.60
	78 865	28 810	47 260	29.50	53.05
Plain 1-in. Round, Annealed	31 730	25 080	45 120	30.00	52.30
	39 809	26 060	45 250	31.75	53.80
Threaded 1-in. Round	2 764	30 950	51 060	24.25	37.10
	2 607	31 930	50 770	21.25	38.91
Threaded 1-in. Round, Annealed	4 536	28 260	46 720	28.00	45.90
	4 430	28 430	47 040	30.50	44.20
in. Round, Annealed, Threaded	2 625	29 310	50 930	24.00	31.50
	....	29 200	51 110	24.25	27.70

The following is the result of the letter ballot vote of the committee consisting of 51 members, on the recommendations made in this report:

Items	Affirmative	Negative	Not Voting
I. PROPOSED REVISION OF TENTATIVE STANDARD			
1. Tentative Specifications for Hollow Staybolt Iron (A 86 - 21 T)	23	0	0
II. PROPOSED REVISION OF STANDARDS			
2. Standard Specifications for Welded Wrought-Iron Pipe (A 72 - 24)	23	0	0
3. Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 - 24)	23	0	0
4. Standard Specifications for Staybolt, Engine-Bolt and Extra-Refined Wrought-Iron Bars (A 84 - 24)	23	0	0

This report has been submitted to letter ballot of the committee, which consists of 51 members, of whom 23 have voted affirmatively, none negatively, and 28 have refrained from voting.

Respectfully submitted on behalf of the committee,

H. J. FORCE,
Chairman.

C. C. OSTERHOUT,
Secretary.

EDITORIAL NOTE

The proposed revisions of the Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes; for Welded Wrought-Iron Pipe; and for Staybolt, Engine-Bolt and Extra-Refined Wrought-Iron Bars referred to in this report were accepted for publication as tentative and appear on pages 896 to 897.

The proposed revisions of the Tentative Specifications for Hollow Staybolt Iron were accepted. The specifications as thus revised appear on page 534.

REPORT OF COMMITTEE A-3

ON

CAST IRON

The outstanding item of the work accomplished by Committee A-3 during the year is the decision to recomend that the dimensions of the "arbitration bar," which has been the standard of the Society since 1905 be changed. This is the result of a long-felt desire on the part of those who test cast iron that the arbitration bar should be more sensitive to deflection requirements. A series of tests made by a committee of the American Foundrymen's Association confirmed the action of the former International Committee on Cast Iron, which advanced the proposal of an international export test bar a decade ago, this bar being the American arbitration bar slightly reduced in diameter (from 1.25 in. to 1.20 in.) to conform to metric measurement possibility, and lengthened by 6 in. so that the supports, on transverse test, would be 18 in. instead of 12 in. apart.

At the time this bar was proposed it was intended for the export business only of the countries adopting it. Since that time, however, a closer contact of the testing experts of the countries interested has brought about a desire to extend an international method of testing to the home requirements themselves. At the Paris meeting of foundrymen in 1923, the subject of international action on testing cast iron was reopened and much has been done since by way of progress. Perhaps the greatest difficulty met with lies in the conception of what the testing of a bar of iron shows the engineer in regard to the value of the castings. In other words, does a test bar represent the castings themselves, or only the quality of the metal going into them? This divergence of thought on the subject is brought out forcibly by the recent action of the British Institute of Foundrymen, who hold that a bar represents the casting. Since they could not provide standard bars for every section thickness met with in a casting, they have selected three diameters of bars, one very small, the next is the proposed international test bar, and the third a large diameter bar to represent very heavy castings.

A similar view is held in Scandinavia, where a study of the situation by the chairman of Committee A-3 last winter indicated that the engineers want definite knowledge of the strength of cast iron in every part of a casting for purposes of design, and expect to get it by means of test bars. Unfortunately, knowledge of the peculiar properties of cast iron—in contradistinction to those of steel, brass, aluminum and other metals and alloys that are homogeneous in structure—has not spread far enough as yet for engineers using that material to realize that they are dealing with all varieties of physical structure in the same casting, complicated by all manners of casting strains, which separately or combined may nullify any conclusions predicated upon test bar results.

The American view of the situation, therefore, which is shared by the engineers and foundrymen of Germany, is that test bars simply give information on the quality of the iron going into castings, and that with definite quality requirements for the different classes of castings fixed by specification, and first-class foundry practice, this is all that can be expected in dealing with so unhomogeneous but nevertheless highly useful a material as cast iron.

While, therefore, the committee does not share the British ciew-point of selecting three bars of different diameters to actually represent castings—a thing tried out in Germany many years ago and abandoned as unpractical, and replaced by one bar 1.20 in. in diameter and broken on supports 24 in. apart—the committee does agree with them in holding that the quality of gray cast iron in very heavy work should not be judged by a bar so small in diameter that it would normally have a white fracture. The fact that specifications for very heavy castings, as also for very light ones, do not include the use of test bars with us here at all is the reason why we are perfectly satisfied to work with one bar only, and designate the physical requirements of this bar to be such that we get the kind of iron we want in our castings. It is to be hoped that continued discussion and explanation will lead to a better understanding of the subject at issue, so that the two diverging points of view may be brought into harmony and practical application.

Further work has been done by the A.F.A. Committee on the French method of testing cast iron by the removal of very small bars from the castings themselves. This interesting procedure has, however, not yet been studied sufficiently to be reported on here.

With the adoption of the proposed change in the arbitration bar by the committee, there will be involved corresponding changes in the Standard Specifications for Gray-Iron Castings (A 48 - 18), High-Test Gray-Iron Castings (A 88 - 24), Locomotive Cylinders (A 45 - 14), and Soil Pipe and Fittings (A 74 - 18). These changes are now being worked out, and will be sent out for study and test so that suitable specifications may be presented to the Society for acceptance as tentative in due course.

The committee wishes to report further that through the cooperation of the Bureau of Standards, there has been made a revision of the Standard Methods of Sampling and Analyses of Pig and Cast Iron (A 64 – 16), report of which has been distributed to those interested and will after study and trial be presented to the Society for adoption as tentative to replace the present requirements. The Bureau of Standards is at work on better and more readily applied methods for the determination of oxygen as well as combined carbon in cast iron, so that foundry and machine shop may profit by what can be learned through the use of these methods.

Progress is being made in the matter of preparing a Recommended Practice for the Analysis of the Different Classes of Castings, the standardization of Pattern Work practice, and the unification of the different specifications for cast-iron pipe, with provision for the new development in centrifugal casting of this material.

This report has been submitted to letter ballot of the committee, which consists of 64 members, of whom 33 have voted affirmatively, none negatively, and 31 have refrained from voting.

Respectfully submitted on behalf of the committee,

RICHARD MOLDENKE,
Chairman.

G. C. DAVIES,
Secretary.

DISCUSSION

Mr. Stanley G. Flagg, Jr.[1]—I should like to ask Mr. Moldenke if the committee has done any work on pearlitic cast iron. It seems to me, from what little I know of the subject, that perhaps it would dispose of some of these questions of the varying grain size, especially in different sized castings with the same silicon content. **Mr. Flagg.**

Mr. Richard Moldenke[2] (*Chairman of Committee A-3*)—I suppose it would be well to explain what is meant by "pearlitic" cast iron. You know that the character of the casting of gray iron depends in a great measure upon the nature of the mold that the iron has been poured into. In other words, the rate at which the molten iron loses its heat affects the character of the iron that is in the casting because of the extent of the change of combined carbon to graphite. Now, in the range of casting practice we have as the extreme on one side the pouring of molten iron into chills, that is, under conditions which mean that the iron gets the quickest possible cooling, for such castings as chilled rolls, rock crusher parts, which we deliberately want to be of very hard iron. The next is a green sand mold. That is a mold made of molding sand which is moist. This will cool the molten iron slower than a chilled mold, but still fairly rapidly. Next in progression comes the dry sand mold, which is a carefully dried green sand mold. You thus have the progression of the chilled, the green sand, the dry; and now comes a further step in a mold brought out in Germany during the War. A sand mold is taken and heated almost to redness and poured. As a consequence, this iron will stay liquid a long time and cool very very slowly. So when you pour the same iron into each of these four molds, you get different results—from very hard, chilled iron to very soft. The outcome of the development is that if you take iron with silicon below 1.00 per cent, which would be perfectly white if cast in chills, and pour it into such a heated sand mold you would get a perfectly gray casting, which shows the effect of a long cooling, which allows an iron with very little silicon to give a metal of very fine microscopic structure, which is entirely pearlitic, and which works magnificently under wear. We have here, unfortunately, a new name for a variety of cast iron, but it is a good material. I want to say, however, that when in Germany last winter, I **Mr. Moldenke.**

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[1] President, Stanley G. Flagg and Co., Inc., Philadelphia.

[2] Consulting Metallurgist, Watchung, N. J.

Mr. Moldenke.

inquired into what had become of this pearlitic iron, and found that for light and medium castings splendid results were had, but with heavy sections the pearlitic structure was not obtainable. Pearlitic cast iron, therefore, seems adaptable for light and medium castings, but not for heavy ones.

Mr. Edwards.

Mr. E. J. Edwards.[1]—In regard to the first sentence of the report of Committee A-3, I would suggest that it be changed slightly to state that the question of a new arbitration test bar is being given consideration by the committee. As I understand it, this question of an arbitration test bar must go back to the several sub-committees in charge of specifications before it will be acted upon by the general committee. Unless there is some good reason for it, I personally believe that we should go slow in changing the arbitration test bar.

Mr. Moldenke.

Mr. Moldenke.—Committee A-3 has voted that they desire to have the change. While the committee has so voted, the Society has not acted upon the matter. To carry out this change it will be necessary to change four individual specifications. So it is not settled yet, by any means.

Mr. Edwards.

Mr. Edwards.—I am not opposed to the changing of the arbitration bar if there is some good reason for it. As I understand it, we have no definite knowledge that the European nations are going to adopt this bar. In this country the 12-in. bar has been used for a great many years. There are a large number of testing machines in use throughout the country that cannot be changed, that are built especially for making a transverse test on cast iron. Unless the committee can point out some specific reason why we should make that change, we should go slow. No doubt, the 12-in. bar would give us as much information as a longer bar.

Mr. Moldenke.

Mr. Moldenke.—The question of the international aspect of this is only incidental. The real reason is that we want some bar which will give us more light on the bending qualities of cast iron. Now, the bar that would bend 0.1 in. with 12 in. between supports, may bend 0.27 in. with 18 in. between supports. Hence you have a much greater range of bending data to work with. Take, for instance, the test bar for cast-iron pipe, that sometimes bends as much as 0.47 in. You really learn a good deal from such a bar. The idea of the change in the arbitration bar length isn't so much to favor international action, because that may not come for some time, but we feel the arbitration bar is too short to give us the information we want.

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[1] Engineer of Tests, American Locomotive Co., Schenectady, N. Y.

MR. J. H. GIBBONEY.[1]—This question of changing the dimensions of the arbitration test bar really has a far-reaching significance in view of the fact that most of the information we now have in this country on cast iron is based on the present bar. With the proposed change we would have an entirely new measuring stick with nothing for ready comparison, unless the committee gives us a conversion formula, so it seems there should be very substantial reasons for making this radical departure to warrant its approval. **Mr. Gibboney.**

MR. EDWARDS.—From the way this report reads, will not the Society expect the committee to offer some definite changes in the several iron specifications? **Mr. Edwards.**

THE SECRETARY-TREASURER.—I do not interpret the report that way. I should say that Committee A-3 is simply telling the Society that it has decided to recommend a modification of the arbitration test bar; it may next year propose specific revisions in the iron specifications or it may reconsider the entire matter. The acceptance of this report does not constitute acceptance of the modified test bar or instructions to Committee A-3 to propose revisions of the cast iron specifications next year. Of course, if the committee adheres to its decision to modify the bar, the several specifications using this bar must be revised in order to make the revision effective. **Secretary-Treasurer.**

MR. J. T. MACKENZIE.[2]—The proposed new arbitration bar makes it possible to bring the cast-iron pipe tests into line with other cast-iron products. The old bar was much too rigid for use in this industry, but I think the new one will be very satisfactory. **Mr. MacKenzie.**

THE SECRETARY-TREASURER.—Mr. Gibboney's point, I think, is a good one. Will Committee A-3, if it asks the Society to make these proposed changes, also give us a suitable conversion table, so that data from the new 18-in. bar can be intelligently compared with the existing data from the present 12-in. bar? **Secretary-Treasurer.**

MR. MOLDENKE.—Yes, that is the situation. We will probably have a factor which can be applied to the results from the old method. That is exactly what happened in 1905. Nearly everybody then used a 1-in. square test bar and it took many years to get around to the $1\frac{1}{4}$-in. round bar, so it was necessary for a long time to use a factor for the conversion of results. We will take care of this situation. **Mr. Moldenke.**

MR. T. H. WIGGIN[3] *(by letter)*.—As a member of Committee A-3 on Cast Iron the writer voted "Yes" on the approval of the current report which is mainly devoted to recommending a change in the **Mr. Wiggin.**

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[1] Chief Chemist, Norfolk and Western Railway Co., Roanoke, Va.

[2] Chief Chemist, American Cast Iron Pipe Co., Birmingham, Ala.

[3] Consulting Engineer, New York City.

Mr. Wiggin. dimensions of the arbitration bar from 1.25 in. to 1.20 in. in diameter and from 12 in. to 18 in. in span for transverse tests. The report also states anew the century old question of direct *versus* indirect tests of cast iron.

As things stand the proposed change is a logical one. The increased span for transverse tests permits easier measurement of differences in deflection and the change in diameter makes the bar like one of the three British Standard bars and brings it into conformity with Continental practice as to indirect testing.

The writer has been a student of the testing of cast iron for about thirty years and in former times gave much attention at the foundry and elsewhere as to the relation between metal in test bars and metal in castings, particularly pipe and valve castings. The indirect testing of cast iron by bars of one size, such as the arbitration bar, is fairly satisfactory for the metallurgist at the foundry who knows the chemistry of his cupola charges and can trace the effect on the test bars and castings of day by day variations in the charging. The test bar of one size theoretically might also guide an intelligent inspector if he would study results in connection with his observations on the castings which he is inspecting and of which he makes an occasional rejection. He may witness the breaking up of these rejected castings. An inspector with resourcefulness of this kind does not remain long an inspector in a subordinate position where he has time for such observations.

Since irons of quite different texture and composition not infrequently give the same results in test bars of constant size, the purchaser, sitting in an office in some city far from the foundry, can have little certainty from the reported results of test bars of one size. If he also had a chemical analysis and was skilled in interpretation he could feel reasonably well satisfied. If, as is usual, he has only a casual knowledge of metallurgy he will be better served by the English system of test bars adapted to thickness of casting and still better served by samples cut from the casting if such samples can be obtained without hurting the casting for its use.

In 1899, the writer suggested the adoption of a 3-size system of test bars similar to the present British standard. Recently, for cast-iron pipes, which are peculiarly amenable to direct testing, he has been advocating the direct test afforded by testing rings cut from the pipe. Some investigation of this possibility has been made by certain pipe foundry men.

It is the opinion of the writer that Committee A-3 has much to do in painstaking investigation of this old question of direct *versus* indirect testing of cast iron. There is not enough exact data available

Mr. Wiggin. to users as to the chemical composition and strength, in castings themselves, of cast iron producing given results in test bars. An indirect test ought to be definite. The writer has not found the single size of bar at all definite nor a real safeguard. International uniformity in so incomplete a means of defining a structural material will doubtless aid in certain comparisons made by foundry experts but will not solve the problem for the user.

Mr. Moldenke. MR. MOLDENKE (*closure by letter*).—Mr. Wiggin is entirely right in his desire for a *direct* test for judging the value of castings. But just how to make such tests for certainty in results has yet to be shown. Cutting pieces from the castings where it will not matter does not answer. This is the French method now being advocated in connection with international standard methods of testing cast iron. Such a test gives the value of the metal only for the point of fracture. It may have an entirely different value at other points of the casting, depending upon the thickness, position in section, shrinkage of the iron, castings strains and other considerations. Merely to get a line on the weakest metal in the casting in order to play safe is simply begging the question, and the chances of working out a test of this kind satisfactory to the foundryman who knows what cast iron is, and the purchaser who knows what properties he wants in his castings are not at all bright. The next best method is to test each casting under an overload and observe results. This is done in part with cast-iron pipe, water under a pressure of 50 per cent over the working load being required to be applied successfully. Why not carry this idea further and support the length of pipe at the ends, load it in the middle and require a minimum strength and deflection program as dictated by experience? The hammer test, hydraulic test and such a bending test on the full sized casting should obviate the necessity of a test bar, even though this does not give a line on the ultimate strength of the metal itself.

In the chilled cast-iron car wheel we have another example of getting near the direct test, for here a few specimens of a batch of castings are tested to destruction, and the rest of the batch—all made under conditions as nearly identical as may be possible—are judged accordingly. There are, however, not many lines of castings that can be tested in this manner, and hence the demand for a method that is more generally applicable where castings are to be made under specification at all. The test bar was the logical development of this desire, and from being attached to the casting originally, it has come to be separately made and under conditions insuring the fairest possible chance to become a normal casting.

Mr. Moldenke. Mr. Wiggin is correct in calling the use of the test bar an *indirect* test, and the question of this test as against a *direct* one is without doubt a very old one. Until rather recently, however, the question of whether the test bar represents the castings or only the metal in them, had not become acute. The impetus given to research work, with the awakened interest in specification making—national as well as international—has brought out the mooted question very prominently. The American viewpoint that the test bar merely gives information on the quality of the metal going into the castings themselves is championed by this Society and the American Foundrymen's Association—both qualified bodies in the premises. The other viewpoint will be found prevalent amongst European engineers interested in the foundry product. The European foundryman, when closely pressed in argument on this point, invariably admits that the American contention is the correct one, but that circumstances—meaning thereby engineering specifications—demand it otherwise. As cast iron research work progresses in the world, this question will settle itself in due course.

The selection of three standard bars, as mentioned by Mr. Wiggin, is really an attempt to make a small-diameter bar represent light castings, a medium-size bar, medium thickness work, and the large-diameter test bar, heavy work. The Institute of British Foundrymen has adopted this system, and it remains to be seen how it works out in England. Germany had tried the same thing some years ago but abandoned the idea, coming back to one standard bar of the same diameter as the middle British one, but longer. Evidently they found no special need for the other two sizes. Indeed, the general opinion in Great Britain is that the middle bar will be the one most used.

Both Mr. Wiggin and the British Foundrymen are, however, right on one point, and that is the incorrectness of making the results of a single diameter bar serve to base judgment on castings of all kinds of thickness. This, whether the American or European view be held. But if this is so, why stop then at only three thicknesses as measures. Undoubtedly it is true that an iron suitable for heavy chilled crusher parts would be white in the test bar and gray in the iron back of the chilled portions of the casting. The results of a white test bar cannot surely be used to judge the quality of the cast iron which is gray in such a large casting. This is an argument brought by our British friends against the American conception of the functions of the test bar. The reply we give is that the single test bar is a matter of expediency simply. We, over here, so far, have no heavy castings to make under specifications which would mean a white iron test bar.

Mr. Moldenke.

When that time comes we will be glad to adopt the heavy British bar, but only for special purposes, and with the distinct understanding that the bar simply represented the metal in the casting and not the casting itself. In other words, we do not wish to burden our specifications with items applicable only to very special work. Indeed, it will be noted that we have only two general cast-iron specifications, one for the ordinary run of specification castings and the other for the high-test run of specification castings. Special lines of work, such as car wheels, pipe, cylinders, etc., have their special sets of specifications which prescribe widely differing lines of tests.

In conclusion, may I say that the very impetus given to cast-iron research work above mentioned is the reason for the action of Committee A-3. Abundant study and testing of cast iron generally in this country has shown that the arbitration bar is too short to give satisfactory deflection results. Hence, we desire a change, and the proposed modification is the outcome of this investigation. Specifications, if anything, should be in advance of general practice, so that laggards must needs improve their operating methods. It is desirable, of course, to make such changes as easily assimilable as possible, and no doubt ways and means will be duly found to adjust the testing machines in existence, as well as to find factors of conversion in results between the old and the new bars.

REPORT OF COMMITTEE A-4

ON

HEAT TREATMENT OF IRON AND STEEL

The committee has held two meetings since the last Annual Meeting. It has reported to the Executive Committee of the Society that it does not approve the proposed definitions of heat treatment terms submitted by the American Society for Steel Treating for the reasons given in a special report.

The committee has approved for acceptance by the Society for publication as tentative, a new Recommended Practice for Carburizing and Heat Treatment of Carburized Objects, which is appended hereto,[1] to supersede, when adopted, the present Recommended Practice for Heat Treatment of Case-Hardened Carbon-Steel Objects (A 37 - 14).[2]

This report has been submitted to the letter ballot of the committee, which consists of 43 members, of whom 35 have voted affirmatively, 1 negatively, and 7 have refrained from voting.

Respectfully submitted on behalf of the committee,

H. M. Boylston,
Chairman.

J. H. Hall,
Secretary.

Editorial Note

The proposed revision of the Recommended Practice for Heat Treatment of Case-Hardened Carbon-Steel Objects was accepted for publication as a new tentative recommended practice and appears on page 568.

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[1] See p. 568.—Ed.

[2] 1924 Book of A.S.T.M. Standards.

REPORT OF COMMITTEE A-5
ON
CORROSION OF IRON AND STEEL

The report of Committee A-5 consists mainly of the reports of its sub-committees, which have been active and have made definite progress in the work during the past year.

Sub-Committee III on Inspection of the Fort Sheridan, Pittsburgh, and Annapolis Tests, reports a few additional failures at the Fort Sheridan and Annapolis locations (the Pittsburgh tests being completed in 1923), the rate of corrosion at these two locations continuing very slow. With two exceptions, the failures at these stations are confined to the light gage non-copper-bearing groups, several entire groups of non-copper bearing metals having failed at Fort Sheridan.

Sub-Committee V on Total Immersion Tests reports additional failure at Annapolis and Washington locations and presents tables giving the record of failures at these points. It is noted that the light gage sheets at Annapolis immersed in the brackish water of the Severn River show approximately 40 per cent longer service than the same group of sheets exposed in Washington City water and about 26 times the service life of sheets immersed in mine water. The sub-committee has completed arrangements for extending the tests to include immersion in ocean water at Portsmouth, N. H., and Key West, Fla.

Sub-Committee VI on Specifications for Metallic-Coated Products has been investigating the advisability of revising the Tentative Specifications for Hot-Dipped Galvanized Sheets (A 93 - 24 T) to extend the list of gages of Class A, Table I, sheets carrying a minimum of 2.5-oz. coating to include gages lighter than No. 22. However, the information so far developed indicates that considerable difficulty is experienced in applying a uniform 2.5-oz. coating to sheets lighter than No. 22 gage, and therefore recommends that no change in the tentative specifications be made at this time. The sub-committee has made considerable progress in the preparation of specifications for zinc-coated wire and sherardized materials, and expects to offer additional specifications in another year.

Sub-Committee VII on Accelerated Tests has arranged to conduct a series of tests on specimens similar to the types to be included in the Field Tests now being arranged by Sub-Committee VIII, this testing of the tests being preliminary to its comprehensive study of specimens cut from the same products included in the service exposure tests. Two new tests for the laboratory examination of zinc-coated materials are presented as information.

Sub-Committee VIII on Field Tests of Metallic-Coated Products reports very substantial progress in developing the details of its comprehensive exposure tests which will include hot-dipped galvanized sheets of five different weights of coating, the base metals being non-copper-bearing and copper-bearing commercial open-hearth steel and pure iron, zinc-coated wire of four weights of coating on the Common, Siemens-Martin and Extra-High-Strength strands and applied by the hot-dipped process as ordinarily applied, and as modified to give a preliminary carbonization, and a subsequent heat treatment or annealing. Chain link fencing of two weights of coating will be tested, as well as structural shapes, hardware, and line materials coated by different processes. This test is attracting widespread interest since there is apparently little authentic comparative data on the relative resistance of various metallic coatings under the different atmospheric conditions in this country. It is gratifying to report that some sixty companies have contributed money and materials and that such support has made the investigation possible. It is anticipated that the tests will be exposed at the five locations, namely, Pittsburgh, Pa., Altoona, Pa., State College, Pa., Sandy Hook, N. J., and Key West, Fla., during the coming summer. The sub-committee expects to present with its next report an historical study of the development of the methods of coating iron and steel with various non-ferrous metals, such as zinc, lead, tin, and cadmium.

This report has been submitted to letter ballot of the committee, which consists of 69 members, of whom 55 have voted affirmatively, none negatively, and 14 have refrained from voting.

Respectfully submitted on behalf of the committee,

J. H. GIBBONEY,
Chairman.

JAMES ASTON,
Secretary.

REPORT OF SUB-COMMITTEE III ON INSPECTION OF THE FORT SHERIDAN, PITTSBURGH, AND ANNAPOLIS TESTS

Sub-Committee III on Inspection has held two meetings during the past year.

First Meeting

The first meeting was held at Annapolis, Md., on October 23, 1924, with Messrs. Aupperle, Cooper, Fleming, Gibboney, McDonnell, Smith, Taylerson, and the representative of the Bureau of Steam Engineering of the United States Navy Department present; the representative of the Quartermaster's Corps of the United States War Department being absent.

One additional failure, Sheet S-7 (No. 22 Gage Low-Copper Pure Iron, Series B), manufactured by the American Rolling Mill Co., was recorded, the failure being due to a small hole near the bottom of the sheet. No photographic record was made of this failure.

Sheet V-5 was noted as having received mechanical damage due to shot hole.

The rate of corrosion at Annapolis continues very slow as compared with the results obtained at Pittsburgh and Fort Sheridan. It is also noted that the corrosion at this station is more uniform throughout the sheet, and that in some groups the sheets are becoming very thin.

Mr. Gibboney reported his inspection of the sheets at Fort Sheridan, Ill., on October 12, 1924, with no additional failures since the April 16, 1924, inspection, therefore no inspection at this station was scheduled for the fall.

Second Meeting

The second meeting was held at Philadelphia, Pa., on March 18, 1925, at which time Mr. H. E. Smith reported that he inspected the sheets at Fort Sheridan, Ill., on March 11, 1925, and found one additional failure, Sheet X-16 (Low-Copper Pure Iron, Series B), manufactured by the Inland Steel Co. This sheet showed a corrosion crack about three inches long about transverse of the sheet, and with respect to longitudinal location near the bottom rail of the rack.

This is the fourth failure in this particular group of fourteen sheets. No photographic record was made of this failure.

The sheets at Annapolis, Md., were inspected on March 27, 1925, by Messrs. Cooper, McAdam and Thompson, who report the following additional failures:

Sheet B-4 (No. 22 Gage Open-hearth Steel, Series A), failed due to a hole in the upper left hand corner.

Sheet S-8 (No. 22 Gage Low-Copper Pure Iron, Series B), manufactured by the American Rolling Mill Co. This sheet failed due to a small hole about twelve inches from the top of sheet.

No photographic record was made of the failed sheets at Annapolis.

The sub-committee at its inspection of October, 1924, and March, 1925, observed that the condition of the surfaces of the sheets as regarding texture, color, and adherence of rust, show substantially the same general characteristics as recorded at preceeding inspections, and for this reason detailed data in the form of condensed statements of numerical rating are not made a part of this report.

In view of the fact that only four additional failures were recorded at Annapolis and Fort Sheridan since the last annual report, tables showing the failures at these stations, grouped as regarding the presence of copper, which have formed a part of previous reports, are omitted. It is sufficient to state that at the Fort Sheridan location none of the copper-bearing sheets of No. 16 gage or No. 22 gage have failed after 96 months' exposure, while one of the No. 16 gage and 46 of a total of 83 of the No. 22 gage low-copper bessemer and open-hearth and pure iron sheets have failed, the order of failure paralleling the more rapid results obtained at the Pittsburgh location. At Annapolis, the four failures so far recorded are all of No. 22 gage sheets, two being low-copper pure iron, one low-copper open-hearth steel, and one copper-bearing open-hearth steel.

Respectfully submitted on behalf of the sub-committee,

J. H. GIBBONEY,
Chairman.

REPORT OF SUB-COMMITTEE V ON TOTAL IMMERSION TESTS

Sub-Committee V on Total Immersion Tests reports that the test racks have been periodically inspected. It was reported in 1924 that all the No. 22 gage and all the No. 16 gage samples had failed at the Calumet Mine test and also all that the No. 22 gage samples had failed at the Bureau of Standards Washington test.

At present, 22 samples out of 150 of the No. 16 gage at the Bureau of Standards, Washington, have failed, and 131 samples out of 144 of the No. 22 gage at Annapolis have failed.

Table I gives the record of No. 16 gage samples which have failed at the Bureau of Standards' test racks at Washington, D. C.

Table II gives the record of the No. 22 gage samples which have failed at the Naval Experiment Station at Annapolis.

The Bureau of Standards has not completed the preparation of the samples for the salt-water immersion tests that are to be made with the assistance of the Bureau of Construction and Repair of United States Navy. The samples will be prepared within a few weeks, and the tests will be made at the Navy Yards, Portsmouth, N. H., and at Key West, Fla. It has been decided to weigh the samples before placing in the racks and also after failure.

Respectfully submitted on behalf of the sub-committee,

WILLIAM A. COOPER,
Chairman.

TABLE I.—REPORT ON SUBMERGED TESTS. SHEETS EXPOSED AUGUST 10, 1920, IN RUNNING WASHINGTON CITY WATER AT THE U. S. BUREAU OF STANDARDS, WASHINGTON, D. C.

No. 16 GAGE SHEETS

Group	Designation	Series	Description	Average Analysis, per cent						Number of Sheets in Test	Failures on Date of Inspections						
				Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper		Jan. 24, 1924, 1261 Days	Aug. 14, 1924, 1464 Days	Sept. 20, 1924, 1501 Days	Nov. 20, 1924, 1562 Days	Dec. 17, 1924, 1589 Days	Jan. 14, 1925, 1624 Days	Feb. 28, 1925, 1662 Days
AA	Bessemer Steel	A	Non-Copper-Bearing	0.053	0.428	0.088	0.042		0.009	6		37					
ZZ	Bessemer Steel, Youngstown Sheet and Tube Co	B	" " "	0.076	0.385	0.096	0.037		0.012	6							
II	Bessemer Steel	A	Copper-Bearing	0.055	0.381	0.099	0.066		0.236	6		55					
ZZ1	Bessemer Steel, Youngstown Sheet and Tube Co	B	" "	0.080	0.400	0.091	0.035	0.056	0.154	6						106	106
ZZ2	Bessemer Steel, Youngstown Sheet and Tube Co	B	" "	0.088	0.306	0.089	0.046		0.271	6		213	213				
ZZ3	Bessemer Steel, Youngstown Sheet and Tube Co	B	" "	0.066	0.363	0.085	0.033		0.548	6							314 314
OO	Basic Open-Hearth Steel	A	Non-Copper-Bearing	0.116	0.378	0.010	0.026	0.005	0.030	6	35						36
MM	Basic Open-Hearth Steel, Alan Wood Iron and Steel Co	B	" " "	0.101	0.426	0.009	0.032		0.048	6						39	
ZZ4	Basic Open-Hearth Steel, Youngstown Sheet and Tube Co	B	" " "	0.029	0.395	0.044	0.026		0.029	6		422		427	426		
BP	Basic Open-Hearth Steel	A	" " "	0.043	0.316	0.033	0.062	0.097	0.126	6							
HH	Basic Open-Hearth Steel	A	Copper-Bearing	0.085	0.391	0.013	0.025		0.230	6					39		
TT	Basic Open-Hearth Steel, Newport Rolling Mill Co	B	" "	0.078	0.301	0.008	0.021		0.215	6							
ZZ5	Basic Open-Hearth Steel, Youngstown Sheet and Tube Co	B	" "	0.065	0.367	0.053	0.064		0.223	6							510
ZZ6	Basic Open-Hearth Steel, Youngstown Sheet and Tube Co	B	" "	0.090	0.330	0.034	0.055		0.574	6							615
ZZ7	Basic Open-Hearth Steel, Youngstown Sheet and Tube Co	B	" "	0.063	0.391	0.052	0.045		0.355	6							
CC	Pure Iron	A	Non-Copper-Bearing	0.021	0.033	0.004	0.031		0.060	6							25
DD	Pure Iron	A	" " "	0.021	0.029	0.004	0.025		0.025	6							
SS	Pure Iron, American Rolling Mill Co	B	" " "	0.014	0.018	0.006	0.024		0.027	6							
XX	Pure Iron, Inland Steel Co	B	" " "	0.026	0.054	0.008	0.026		0.029	6					11		
CCC	Pure Iron	A	Copper-Bearing	0.016	0.026	0.005	0.026	0.005	0.220	6							
UU	Pure Iron, Allegheny Steel Co	B	" "	0.016	0.065	0.009	0.030		0.244	6							
VV	Pure Iron, Whitaker-Glessner Co	B	" "	0.017	0.039	0.007	0.021		0.290	6							
KK	Acid Open-Hearth Steel	A	Copper-Bearing	0.118	0.408	0.081	0.043		0.229	6							57
YY	Puddled Iron, Youngstown Sheet and Tube Co	B	Non-Copper-Bearing	0.030	0.060	0.124	0.018	0.216	0.024	6				7			1
EE	Puddled Iron	A	Copper-Bearing	0.033	0.045	0.117	0.017	0.132	0.325	6							
										150	1	4	1	2	3	2	9 22

TABLE II.—REPORT ON SUBMERGED TESTS. SHEETS EXPOSED JUNE 2, 1920, IN RUNNING SEVE

No. 22 GAGE SH

Group	Designation	Series	Description	Average Analysis, per cent. Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Number of Sheets in Test	Oct. 18, 1922, 868 Days	April 14, 1923, 1046 Days	June 8, 1923, 1101 Days	July 11, 1923, 1134 Days	Aug. 17, 1923, 1171 Days	Sept. 17, 1923, 1202 Days	Oct. 18, 1923, 1232 Days
A	Bessemer Steel	A	Non-Copper-Bearing	0.034	0.373	0.093	0.035	0.0065	0.016	6						2	9
Z	Bessemer Steel, Youngstown Sheet and Tube Co.	B	" " "	0.067	0.400	0.104	0.046	0.009	0.013	6					1 2		3
Z1	Bessemer Steel, Youngstown Sheet and Tube Co.	B	" " "	0.077	0.435	0.096	0.036	0.003	0.139	6			102		102 108		102
I	Copper-Bearing Bessemer Steel	A	Copper-Bearing	0.053	0.344	0.096	0.066	0.0035	0.249	6						1	3
Z2	Copper-Bearing Bessemer Steel, Youngstown Sheet and Tube Co.	B	" "	0.060	0.388	0.095	0.041	....	0.266	6		207			204		
Z3	Copper-Bearing Bessemer Steel, Youngstown Sheet and Tube Co.	B	" "	0.051	0.366	0.112	0.052	....	0.541	6							
O	Basic Open-Hearth Steel	A	Non-Copper-Bearing	0.115	0.512	0.010	0.029	0.251	0.014	6		2 5	4 7		3		
B	Basic Open-Hearth Steel	A	" " "	0.034	0.226	0.0115	0.055	0.0045	0.144	6							
Z4	Basic Open-Hearth Steel, Youngstown Sheet and Tube Co.	B	" " "	0.077	0.351	0.068	0.055	0.004	0.185	6		417				413	417
H	Copper-Bearing Basic Open-Hearth Steel	A	Copper-Bearing	0.085	0.367	0.011	0.027	0.0025	0.252	6					2 5		
M	Basic Open-Hearth Steel, Alan Wood Iron and Steel Co.	B	" "	0.099	0.391	0.012	0.042	0.012	0.188	6			4	7	9		13
T	Copper-Bearing Basic Open-Hearth Steel, Newport Rolling Mill Co.	B	" "	0.078	0.322	0.0095	0.019	0.010	0.237	6		24	4		22		2
Z5	Copper-Bearing Basic Open-Hearth Steel, Youngstown Sheet and Tube Co.	B	" "	0.065	0.350	0.047	0.0535	0.003	0.189	6		501				503	503 503
Z6	Copper-Bearing Basic Open-Hearth Steel, Youngstown Sheet and Tube Co.	B	" "	0.060	0.320	0.046	0.064	0.003	0.263	6			603		602		602
Z7	Copper-Bearing Basic Open-Hearth Steel, Youngstown Sheet and Tube Co.	B	" "	0.070	0.415	0.049	0.051	Trace	0.634	6		705			707		
D	Low-Copper Pure Iron	A	Non-Copper-Bearing	0.022	0.024	0.0038	0.024	0.002	0.026	6		1 15				4	
X	Low-Copper Pure Iron, Inland Steel Co.	B	" " "	0.011	0.065	0.006	0.029	0.003	0.033	6		3 12	1				9
S	Low-Copper Pure Iron, American Rolling Mill Co.	B	" " "	0.011	0.015	0.006	0.022	0.007	0.022	6		2 4		6	8 10		
C	Copper-Bearing Pure Iron	A	Copper-Bearing	0.025	0.036	0.0066	0.035	0.002	0.194	6		2 10					
U	Copper-Bearing Pure Iron, Allegheny Steel Co.	B	" "	0.017	0.078	0.008	0.039	0.004	0.253	6							
V	Copper-Bearing Pure Iron, Whitaker-Glessner Co.	B	" "	0.013	0.068	0.007	0.022	0.005	0.306	6		2			8	10	
K	Copper-Bearing Acid Open-Hearth Steel	A	Copper-Bearing	0.111	0.435	0.087	0.045	0.0027	0.220	6					3		
Y	Low-Copper Puddled Iron, Youngstown Sheet and Tube Co.	B	Non-Copper-Bearing	0.030	0.053	0.149	0.021	0.220	0.020	6	1	1 2		2 3			
E	Puddled Iron	A	Copper-Bearing	0.039	0.032	0.114	0.021	0.110	0.241	6							
										144	1	18	7	4	16	6	11

PLATE I.
PROC. AM. SOC. TESTING MATS.
VOL. 25, PART I.
REPORT OF COMMITTEE A-5:
TOTAL IMMERSION TESTS.

SEVERN RIVER WATER AT THE NAVAL ENGINEERING EXPERIMENT STATION, ANNAPOLIS, MD.

AGE SHEETS

Failures on Date of Inspections																	
1232 Days	Nov. 19, 1923, 1264 Days	Dec. 21, 1923, 1296 Days	Jan. 22, 1924, 1328 Days	March 4, 1924, 1370 Days	April 3, 1924, 1400 Days	May 5, 1924, 1432 Days	June 7, 1924, 1465 Days	July 9, 1924, 1497 Days	Aug. 8, 1924, 1527 Days	Sept. 9, 1924, 1559 Days	Oct. 9, 1924, 1589 Days	Nov. 11, 1924, 1622 Days	Dec. 13, 1924, 1654 Days	Jan. 14, 1925, 1686 Days	Feb. 28, 1925, 1721 Days	Average Number of Days	
				4 5		7				12						1360.8	
							4	4 4								1338.8	
		108		108												1223.5	
	15													9	12		
	204				207			207		206						1322.8	
							301			303							
	301	301					305			305						1434.6	
	9															1121.5	
					4	15	16					9					
		416		413	416											1257.7	{ 2 1139 Low Copper 4 1317 High Copper
					14	8 17		11								1350.5	
													11				
					20												
503	501													501		1094.6	
			603								602						
					705		707		707								
								13									
					8									5		1073	
		12														1144	
				5	8			6		12						1319.6	
	3 11		13		5			7 9								1375	
		4	6		12											1240.5	
				6		8		12	10 15							1420.6	
					3											1104.6	
			2	4													
	7	5	4	7	11	5	5	9	3	5	1	1	1	3	1		

REPORT OF SUB-COMMITTEE VI
ON
SPECIFICATIONS FOR METALLIC-COATED PRODUCTS

Sub-Committee VI on Specifications for Metallic-Coated Products has held two well attended meetings. Owing to the largely increased amount of work assigned to the sub-committee, several groups have been appointed for special duties.

The sub-committee has been requested from a number of sources to investigate the advisability of revising the Tentative Specifications for Hot-Dipped Galvanized Sheets (A 93 - 24 T),[1] to extend the list of gages under Class A, of Table I for material carrying a minimum 2.5-oz. coating, to include gages lighter than No. 22. However, the information so far developed apparently indicates that considerable difficulty is experienced in applying a uniform 2.5-oz. coating to sheets lighter than No. 22 gage.

Requirements for a Class D, covering "tight coated" sheets which will stand severe forming, and also for the base metal for galvanized sheets, and for sherardized coatings are being actively studied and it is hoped definite recommendations can be submitted next year.

The sub-committee has prepared a preliminary draft of requirements for galvanized wire, appended hereto, which is offered for study and comment. It is hoped that it may be sufficiently developed in another year for presentation as a tentative standard.

The sub-committee is now considering the omission from the specifications of sheets of gages having odd numbers, as proposed by the United States Board on Simplified Practice.

Respectfully submitted on behalf of the sub-committee,

H. E. Smith,
Chairman.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 775 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 52.

APPENDIX

PROPOSED REQUIREMENTS FOR ZINC-COATED OR GALVANIZED WIRE

MATERIAL

Zinc.—The zinc used for galvanizing shall be high-grade or prime western spelter, complying with the requirements for these grades of the Standard Specifications for Spelter (Serial Designation: B 6) of the American Society for Testing Materials.[1]

WEIGHT OF ZINC COATING

There shall be four classes of zinc coating as follows:

Class A.—The minimum weight of coating for this class shall be 0.8 oz. per sq. ft. of surface, and shall withstand a minimum of four 1-minute dips by the Preece test, and for round wire shall have a minimum nominal diameter of 0.100 in.

Class B.—The weight of coating for this class shall be from 0.60 to 0.80 oz. per sq. ft. of surface, and shall withstand a minimum of three 1-minute dips by the Preece test, and for round wire shall have a minimum nominal diameter of 0.080 in.

Class C.—The weight of coating for this class shall be from 0.40 to 0.60 oz. per sq. ft. of surface and shall withstand a minimum of two 1-minute dips by the Preece test and for round wire shall have a minimum nominal diameter of 0.060 in.

Class D.—The weight of coating for this class shall be less than 0.40 oz. per sq. ft. of surface and shall withstand a minimum of one dip by the Preece test.

METHOD OF TESTING

The zinc coating shall be tested for quantity by a stripping test and for uniformity by the Preece test.

The stripping test for the weight of zinc in the coating in ounces per square foot of surface shall be performed by the sulfuric acid or antimony chloride stripping test as described in the Standard Methods of Determining Weight of Coating on Zinc-Coated Articles (Serial Designation: A 90) of the American Society for Testing Materials[1] but with the amount of iron in the coating deducted.

The Preece test shall be made as described in the Appendix to the Standard Methods of Determining Weight of Coating on Zinc-Coated Articles (Serial Designation: A 90) of the American Society for Testing Materials.[1]

SAMPLING

All testing and sampling shall be made at the place of manufacture.

One sample sufficient for three each of all tests shall be taken at random from each 5000 lb. of wire, but in no case less than three sets of samples shall be taken to represent the shipment.

Should any sample fail to meet the requirements, two additional tests shall be made on the sample, both of which shall meet the requirements in every respect or the lot represented by the sample shall be rejected.

(Requirements for the physical properties of the coating will be added when the information is available.)

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[1] 1924 Book of A.S.T.M. Standards.

REPORT OF SUB-COMMITTEE VII ON ACCELERATED TESTS

The investigation of different types of accelerated or "short time" corrosion tests has been continued by various members of the sub-committee. These tests are of the following types: repeated immersion, simulated atmospheric, and spray or "mist."

Arrangements had been made to try these tests on various grades of galvanized sheets, good, bad and intermediate. On account of unforeseen difficulties in securing this material, it was not submitted to the members of the sub-committee who had agreed to co-operate in carrying out the tests in time for a report this year.

In order to acquire some experience and skill in conducting the various accelerated tests and in interpreting the test results, arrangements have been made to secure and test a set of coated sheet steel specimens representative of essentially all those types which will be included in the field exposure tests which are being arranged by Sub-Committee VIII on Tests of Metallic-Coated Products. It was felt that preliminary test work of this kind was very desirable before attempting to apply the tests to the samples representative of the field-test material to be submitted by Sub-Committee VIII.

Two new tests for the laboratory examination of zinc-coated materials were brought to the attention of the sub-committee for discussion. The first one, which is essentially a modification of the well-known Preece or copper-sulfate immersion test, consists of 1-minute immersions in a solution of 4 oz. (115 g.) of copper sulfate, 100 cc. of sulfuric acid (66° Baumé) and 1 gal. (3.8 liter) of distilled water. The test is conducted like the Preece test, the number of 1-minute immersions (or fraction thereof) necessary to produce deposition of an *adherent* deposit of copper being used as a measure of the test.

The second test, which is recommended particularly for electroplated zinc coatings, consists in immersion in a solution prepared by adding to 300 cc. of distilled water (kept *cold*), (*a*) 35 g. of glacial acetic acid and (*b*) 10 g. of sodium peroxide, the whole being diluted to 1000 cc. with cold distilled water. The samples are kept immersed continuously until evidence of rusting indicates the "break down" of the coating.

These tests are mentioned in this report for information only, the sub-committee expresses no opinion as to the possible merits or limitations of either.

Respectfully submitted on behalf of the sub-committee,

Henry S. Rawdon,
Chairman.

REPORT OF SUB-COMMITTEE VIII ON TESTS OF METALLIC-COATED PRODUCTS

In October, 1924, a small group of members under Mr. J. A. Aupperle as chairman, launched a campaign to secure funds for conducting field tests on metallic-coated products. The sub-committee is gratified to report that about $10,000 has been subscribed. The list of subscribers as of April 20, 1925, is given herewith:

American Chain Co.
American Malleable Castings Association.
American Metal Co.
American Rolling Mill Co.
American Sheet and Tin Plate Co.
American Steel and Wire Co.
American Zinc Institute.
Babcock and Wilcox Co.
Bethlehem Steel Co.
Birdsboro Steel and Foundry Co.
Byers Co., A. M.
Carson, H. Y.
Central Steel Co.
Crane Co.
Eagle-Picher Lead Co.
Electro Sherardizing Co.
John Finn Metal Works.
General Electric Co.
Hanson and Van Winkle Co.
International Derrick and Equipment Co.
International Harvester Co.
Keystone Steel and Wire Co.
Koppers Co.
Lukens Steel Co.
National Tube Co.
New Jersey Zinc Co.
Newport Rolling Mill Co.
Ohio Brass Co.
Pittsburgh Electro Galvanizing Co.
Reading Iron Co.
Republic Iron and Steel Co.
Robinson and Company, Dwight P.,
Roeblings' Sons Company, John A.,
St. Joseph Lead Co.
Sheet Steel Trade and Extension Committee.
Stanley Works.
Standard Oil Company of New Jersey.
Tennessee Coal, Iron and Railroad Co.
Tidewater Oil Co.
Texas Co., The.
United Alloy Steel Corporation
United States Chain and Forging Co.
Vacuum Oil Co.
Western Electric Co.
Western Union Telegraph Co.
Westinghouse Electric and Manufacturing Co.
Wheeling Steel Corporation.
Wood, Iron and Steel Co., Alan.
York Manufacturing Co.
Youngstown Sheet and Tube Co.

In addition very substantial assistance in labor and material has been offered by the New York Central Railroad, the Pennsylvania Railroad, and the Aermotor Co. towards securing and erecting the test racks.

The following members have offered to supply free of cost certain materials to conform to the sub-committee's specifications for field and laboratory tests:

The American Rolling Mill Co.
The American Sheet and Tin Plate Co.
The Youngstown Sheet and Tube Co.
The Andrews Steel Co.
The United Alloy Steel Corporation.
John A. Roebling's Sons Co.
The Keystone Steel and Wire Co.

The report of a small group under Mr. C. S. Trewin, Chairman, appointed to develop the details of the Field Tests follows:

TEST RACKS

After investigating three types of test racks, a design of rack was decided upon. The posts for the racks for the sheets are to be of reinforced concrete with hot-dip galvanized truss and purlin members all to be held together by hot-dipped galvanized bolts. The truss members are to be set at an angle of 30 deg. with the horizontal. The purlin members for the bay are to be devoted to uncoated sheets to be ungalvanized. The sheets are to be fastened to the purlins by means of the standard strap construction using well-galvanized bolts and nuts. In the case of the uncoated sheets, ungalvanized products are to be used. Between each sheet is to be erected a No. 11 gage 3 in. by 3 ft. 6 in. hot-dipped galvanized iron or steel strip, to which are to be attached bent specimens of the test sheets. These No. 11 gage strips are to be punched for the bolts for attaching bent specimens and for the bolts for attaching the strips with strap to the racks. The bolts, nuts and straps used are to be hot-dipped galvanized.

The racks for the wire products are to have reinforced concrete posts and creosoted wooden members applied at an angle of 45 deg., supported by hot-dipped galvanized braces, fastened with hot-dipped galvanized bolts. The wires are to be attached to the cross-arms by either wooden pins and porcelain insulators or porcelain insulators attached to the cross-arms by hot-dipped galvanized wood screws. An extension of this rack is also to be used for the exposure of the hardware, samples of which are to be attached to suitably arranged creosoted wood strips or hot-dipped galvanized structural members.

For the exposure of woven wire fence products, reinforced concrete posts are to be erected in a line parallel to the two racks, these posts to be on approximately 12-ft. centers. If any fence manufacturer wishes to include in this exposure a standard galvanized post member, these post members will be erected as intermediate posts in the fence line, provided two such posts are supplied for each location of test rack.

The spacing in the clear between the parallel racks shall be 8 ft. with a 5-ft. aisle between the ends of the outside racks and any enclosing fence. It has been roughly estimated that the area required for these test racks will be in the neighborhood of 150 ft. square.

LOCATION OF TEST RACKS

The sub-subcommittee, after carefully canvassing the producers and consumers, including the Army and Navy Departments, reported on the advisability of locating the test racks at the locations given below. These locations were approved by the sub-committee and the chairman was authorized to make final arrangements with the proper authorities at each location.

1. A tropical location which will represent conditions met with in practically all the tropical countries or sections where coated articles are used. The offer by the Bureau of Yards and Docks of the U. S. Navy Department of a location on the submarine base at Keywest, Fla., was accepted.

2. A seacoast location representing conditions likely to be met with on the Eastern Seaboard of the United States. The advice of the Navy Department

and of the Coast Artillery to locate on the Proving Grounds at Fort Hancock, Sandy Hook, N. J., was accepted pending official authorization from the Adjutant General of the United States Army.

3. A location representing the maximum of bad manufacturing atmosphere was decided to be filled by a location to be supplied by the U. S. Bureau of Mines in the Pittsburgh district.

4. A location representing manufacturing conditions midway between the Pittsburgh district and a country location free from manufacturing fumes, was offered by the Pennsylvania Railroad at Altoona, Pa., and was accepted.

5. A location in the country giving very pure and comparatively dry atmosphere was discussed and the location at Pennsylvania State College, State College, Pa., was approved.

GALVANIZED SHEETS

Size of Sheets and Test Specimens.—The sheets are to be No. 22 gage, 26 in. wide after corrugation and 9 ft. long. After galvanizing the sheets are to be stamped *T* and *B*, the end marked *T* to be that end first emerging from the galvanized bath, that marked *B* last emerging from the galvanizing bath. They are to be cut and sampled before corrugation as indicated in Fig. 1, starting at the top end of the sheet, the first 3 in. to be rejected, the next 3 in. for laboratory, spot and bend tests, the next 30 in. to be corrugated with standard 2½-in. corrugations and exposed on the test rack. The next 9 in. are to be used for bend tests as described under "Bent Specimens," and exposed on the test rack. The next 3 in. are to be used for laboratory, spot, and bend tests. The next 24 in. are to be used for accelerated tests. The next 30 in. are to be corrugated and exposed on the rack. The next 3 in. are to be used for the laboratory, spot and bend tests, and the end 3 in. are to be rejected.

Base Material.—The base material shall be representative commercial steel and iron made by the basic open-hearth process. Two grades of each type of material shall be used, one containing a maximum of 0.05 per cent copper and the other a minimum of added copper of 0.20 per cent. The limits of composition of the base material (ladle test) in addition to the above shall be:

Carbon	Under	0.20	per cent
Manganese	"	0.60	"
Sulfur	"	0.06	"
Phosphorus	"	0.10	"

The amount of ingot to be cropped is to be left to the judgment of the manufacturer of the base material.

Zinc.—The zinc used for the sheets is to be in accordance with requirements for Prime Western Zinc of the Standard Specifications for Spelter (B 6). Tin may be added if desired by the galvanizer. Aluminum must be absent.

Weight of Coating.—There shall be five weights of coating: Weights of 2½, 2 and 1½ oz. per sq. ft., described as Class A, B, and C, in Table I of the Tentative Specifications for Hot-Dipped Galvanized Sheets; a coating of minimum average weight of 1¼ oz. per sq. ft. (the variations in the weight of coating of the spot tests of these sheets shall correspond to those described in the Tentative Specifications for Hot-Dipped Galvanized Sheets); and a weight of coating from ¾ to 1 oz. per sq. ft. to be known as tight coated material.

Erection of Specimens on Test Rack.—There are to be erected on each test rack the two 30-in. corrugated sections of two sheets of each of the above weights of coating on each of the four bases, making two specimens from 40 sheets. In addition there are to be erected one 30-in. corrugated section of two uncoated sheets of each base material, making eight in all.

Bent Specimens.—It is the opinion of the committee that the exposure of severely bent specimens should be included. Provision has been made for a specimen 9 in. wide by approximately 30 in. long to be cut from near the center of each of the test sheets, as shown in Fig. 1. This strip is to be cut in the direction of rolling into sections 4 in. wide. These are to be formed through the center into a vertical fold by means of a brake. This fold shall be over the following thicknesses of No. 22 gage metal: 0, folded on itself, 1 thickness, 2 thicknesses, 3 thicknesses, 4 thicknesses, 8 thicknesses.

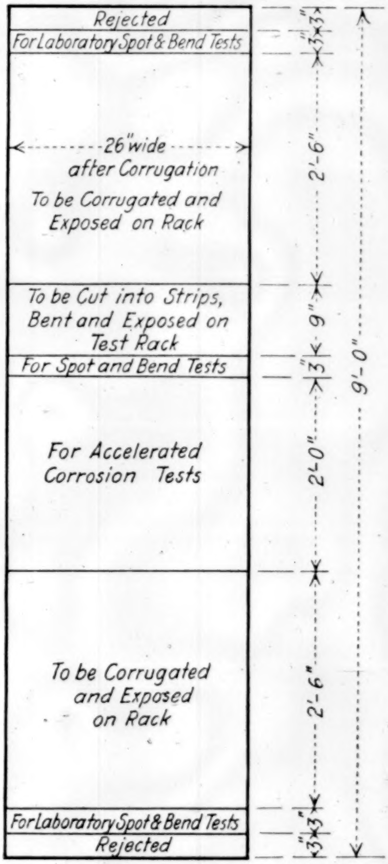
Rejected
For Laboratory Spot & Bend Tests
26" wide after Corrugation
To be Corrugated and Exposed on Rack
To be Cut into Strips, Bent and Exposed on Test Rack
For Spot and Bend Tests
For Accelerated Corrosion Tests
To be Corrugated and Exposed on Rack
For Laboratory Spot & Bend Tests
Rejected
3"
3"
2'-6"
9"
3"
2'-0"
2'-6"
3"
3"
9'-0"

FIG. 1.

These six samples from each of the sheets exposed are to be erected on the test racks. They are to be attached by means of well galvanized bolts to a

3-in. wide galvanized strip. This strip is to be placed on the rack adjacent to the sheet from which these test specimens were cut as indicated in Fig. 1.

Protection of Edges.—All bare edges of sheet specimens erected on the rack are to be protected at the time of erection by painting with "Hermastic" enamel.

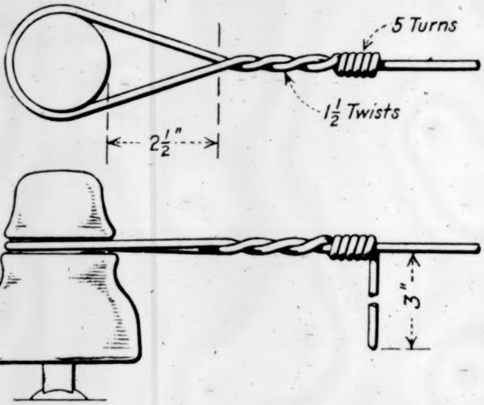

FIG 2.—Western Union Method of Dead Ending No. 8 Iron Wire.

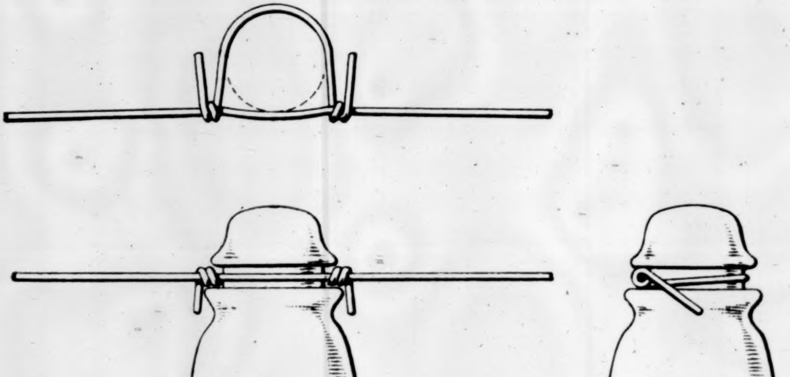

FIG 3.—Western Union Method of Tying Iron Wire to Side Grove Insulator.

WIRE AND FENCING

A small group under Mr. H. J. Horn, Chairman, submits the following outline covering the tests for galvanized wire and fencing.

WIRE

The committee recommends that the following 70 samples of strand and wire be exposed. Each sample is to be 21 ft. 6 in. long (the length of two bays of the rack). For strands, the ends are to be attached by means of hot-dipped galvanized thimbles and clips to the wooden cross-arms of the rack.

The center of each sample is to be supported by passing through a split porcelain insulator attached to the cross-arm of an upright. For the line wires, the ends of the wire samples are to be attached to a standard Western Union insulator by means of a Western Union dead end, see Fig. 2. The wire is to be supported in the center by attaching (by means of a Western Union tie), see Fig 3, to an insulator attached to the cross-arms of an upright. The samples are to be spaced 6 in. apart on the cross-arms, nine samples to a side.

STRAND

Samples of the following strands are to be used:

Common Strand.—Tensile strength of wire from 50,000 to 80,000 lb. per sq. in., $\frac{5}{16}$ in. in diameter consisting of 7 wires 0.105 in. ±0.003 in. in diameter after galvanizing.

Siemens-Martin Strand.—Tensile strength of wire 100,000 lb. per sq. in., $\frac{5}{16}$ in. in diameter consisting of 7 wires 0.105 in. ±0.003 in. in diameter after galvanizing.

Extra-High-Strength Strand.—Tensile strength of wire 200,000 lb. per sq. in., $\frac{3}{8}$ in. in diameter consisting of 7 wires 0.125 in. ±0.003 in. in diameter after galvanizing.

WEIGHT OF ZINC COATING

There shall be four weights of coating:

Class A.—Carrying a weight of coating 0.80 to 1.3 oz. of zinc per sq. ft. of surface.

Class B.—Carrying a weight of coating 0.60 to 0.80 oz. of zinc per sq. ft. of surface.

Class C.—Carrying a weight of coating 0.40 to 0.60 oz. of zinc per sq. ft. of surface.

Class D.—Carrying a weight of coating of 0.40 oz. maximum of zinc per sq. ft. of surface.

ZINC

The zinc or spelter used for galvanizing shall conform in all respects to the requirements drawn up by Sub-Committee VI on Specifications for Metallic-Coated Products of Committee A-5.

Common Strand:

Base Material.—1. Basic open-hearth steel without added copper. The analysis of this base material shall be:

Carbon	0.08 to 0.15	per cent
Manganese, max	0.60	"
Silicon, max	0.12	"
Sulfur, max	0.06	"
Phosphorus, max	0.10	"
Copper, max	0.08	"

Samples.—The following samples are to be erected on each test rack:

Class A.—There shall be six samples, two manufactured by the hot-dip process, one manufactured by the hot-dip process and subsequently heat treated, one manufactured by the hot-dip process upon wire given a preliminary carbonization before galvanizing, and two samples electrogalvanized.

Class B.—There shall be four samples, two manufactured by the hot-dip process, one manufactured by the hot-dip process and subsequently heat treated, and one manufactured by the hot-dip process upon wire given a preliminary carbonization before galvanizing.

Class C.—There shall be four samples, two manufactured by the hot-dip process, and two electrogalvanized.

Class D.—There shall be two samples manufactured by the hot-dip process.

2. Basic open-hearth steel with added copper. The analysis of the copper-bearing open-hearth steel shall be:

Carbon	0.08 to 0.15	per cent
Manganese, max	0.60	"
Silicon, max	0.12	"
Sulfur, max	0.06	"
Phosphorus, max	0.10	"
Copper, min	0.20	"

Samples.—The following samples are to be erected on each rack:

Class A.—There shall be four samples, two manufactured by the hot-dip process, and one manufactured by the hot-dip process upon wire given a preliminary carbonization, and one hot-dipped with a subsequent heat treatment.

Class B.—There shall be four samples, two manufactured by the hot-dip process, one hot-dipped upon wire given a preliminary carbonization, and one by the hot-dip process with subsequent heat treatment.

Class C.—There shall be two samples manufactured by the hot-dip process.

Class D.—There shall be two samples manufactured by the hot-dip process.

Siemens-Martin Strand:

Base Material.—The base material shall be basic open-hearth steel of the following analysis:

Carbon	0.30 to 0.60	per cent
Manganese	0.30 to 0.80	"
Silicon, max	0.15	"
Phosphorus, max	0.06	"
Sulfur, max	0.06	"

Samples.—The following samples are to be erected on each rack:

Class A.—There shall be three samples manufactured by the hot-dip process. No samples of any other class.

Extra-High-Strength Strand:

Base Material.—The base material shall be both basic and acid open-hearth steel of the following analysis:

Carbon	0.60 to 0.90	per cent
Manganese	0.30 to 0.80	"
Silicon, max	0.20	"
Phosphorus, max	0.05	"
Sulfur, max	0.05	"

Samples.—The following samples are to be erected on each rack:

Class A.—There shall be two samples manufactured by the hot-dip process from wire made of basic open-hearth steel and two samples manufactured by the hot-dip process from wire made of acid open-hearth steel. No samples of any other class.

WIRE

The wire shall be 0.165 in. ± 0.004 in. in diameter after galvanizing.

Weight of Zinc Coating.—There shall be four weights of coating:

Class A.—Carrying a weight of coating of 0.80 to 1.3 oz. of zinc per sq. ft. of surface.

Class B.—Carrying a weight of coating 0.60 to 0.80 oz. of zinc per sq. ft. of surface.

Class C.—Carrying a weight of coating 0.40 to 0.60 oz. of zinc per sq. ft. of surface.

Class D.—Carrying a weight of coating 0.40 oz. maximum of zinc per sq. ft. of surface.

Zinc.—The zinc or spelter used for galvanizing shall conform in all respects to the requirements drawn up by Sub-Committee VI on Specifications for Metallic-Coated Products of Committee A-5.

Base Material.—1. Basic open-hearth steel without added copper. The analysis of the base material shall be:

Carbon	0.08 to 0.15	per cent
Manganese, max	0.60	"
Silicon, max	0.12	"
Sulfur, max	0.06	"
Phosphorus, max	0.10	"
Copper, max	0.08	"

Samples.—The following samples are to be erected on each rack:

Class A.—There shall be four samples, two manufactured by the hot-dip process, one manufactured by the hot-dip process and subsequently heat treated, and one manufactured by the hot-dip process upon wire given a preliminary carbonization before galvanizing.

Class B.—There shall be four samples, two manufactured by the hot-dip process, one manufactured by the hot-dip process and subsequently heat treated, and one manufactured by the hot-dip process upon wire given a preliminary carbonization before galvanizing.

Class C.—There shall be two samples manufactured by the hot-dip process.

Class D.—There shall be two samples manufactured by the hot-dip process.

2. Basic open-hearth steel with added copper. The analysis of this base material shall be:

Carbon	0.08 to 0.15	per cent
Manganese, max	0.60	"
Silicon, max	0.12	"
Sulfur, max	0.06	"
Phosphorus, max	0.10	"
Copper, min	0.20	"

Samples.—The following samples are to be erected on each rack:

Class A.—There shall be four samples, two manufactured by the hot-dip process, and one manufactured by the hot-dip process upon wire given a preliminary carbonization, and one hot-dipped with a subsequent heat treatment.

Class B.—There shall be four samples, two manufactured by the hot-dip process, one by the hot-dip process with subsequent heat treatment, and one hot-dipped upon wire given a preliminary carbonization.

Class C.—There shall be two samples manufactured by the hot-dip process.

Class D.—There shall be two samples manufactured by the hot-dip process.

3. Basic open-hearth commercial iron. The analysis of this base material shall be:

Carbon, max	0.06	per cent
Manganese, max	0.15	"
Silicon	trace	
Phosphorus, max	0.04	"
Sulfur, max	0.04	"
Copper, max	0.05	"

Samples.—The following samples are to be erected on each rack:

Class A.—There shall be four samples, three manufactured by the hot-dip process and one manufactured by the hot-dip process upon wire given a preliminary carbonization.

Class B.—There shall be four samples, three manufactured by the hot-dip process and one manufactured by the hot-dip process upon wire given a preliminary carbonization.

Class C.—There shall be two samples manufactured by the hot-dip process.

Class D.—There shall be two samples manufactured by the hot-dip process.

FENCING

The committee recommends that the following 32 samples of woven field fence and chain link fence be exposed. Each sample is to be attached to upright posts, 1 rod (16½ ft.) apart. Two samples of fencing are to be erected in each bay, one on each side of the posts. Samples of the field fence are to be attached to the posts by stringing out the strand wires, wrapping them around the posts, and tying by means of a Western Union type dead end joint.

The bottom of the fence shall be at least one foot from the ground. One-rod samples of chain link fence are also to be used attached to the posts by means of wires wrapped around the posts.

WOVEN FIELD FENCE

The field fence is to be manufacturers' standard field fence woven after galvanizing, made of No. 9 gage wire 39 in. high, 9 strand wires with 6 in. stay spacing.

Weight of Zinc Coating.—There shall be four weights of coating:

Class A.—Carrying a weight of coating 0.80 to 1.3 oz. of zinc per sq. ft. of surface.

Class B.—Carrying a weight of coating 0.60 to 0.80 oz. of zinc per sq. ft. of surface.

Class C.—Carrying a weight of coating 0.40 to 0.60 oz. of zinc per sq. ft. of surface.

Class D.—Carrying a weight of coating of 0.40 oz. maximum of zinc per sq. ft. of surface.

Zinc.—The zinc or spelter used for galvanizing shall conform in all respects to the requirements drawn up by Sub-Committee VI on Specifications for Metallic-Coated Products of Committee A-5.

Base Material.—1. Basic open-hearth steel without added copper. The analysis of this base material shall be:

Carbon	0.08 to 0.15	per cent
Manganese, max	0.60	"
Silicon, max	0.12	"
Sulfur, max	0.06	"
Phosphorus, max	0.10	"
Copper, max	0.08	"

Samples.—The following samples are to be erected on each rack:

Class A.—There shall be two samples, one galvanized by the hot-dip process with subsequent heat treatment, and one galvanized by the hot-dip process.

Class B.—There shall be two samples, one galvanized by the hot-dip process with subsequent heat treatment, and one galvanized by the hot-dip process.

Class C.—There shall be two samples galvanized by the hot-dip process.

Class D.—There shall be two samples galvanized by the hot-dip process.

2. Basic open-hearth steel with added copper. The analysis of this base material shall be:

Carbon	0.08 to 0.15	per cent
Manganese, max	0.60	"
Silicon, max	0.12	"
Sulfur, max	0.06	"
Phosphorus, max	0.10	"
Copper, min	0.20	"

Samples.—The following samples are to be erected on each rack:

Class A.—There shall be two samples, one galvanized by the hot-dip process with subsequent heat treatment, and one galvanized by the hot-dip process.

Class B.—There shall be two samples, one galvanized by the hot-dip process with subsequent heat treatment, and one galvanized by the hot-dip process.

Class C.—There shall be two samples galvanized by the hot-dip process.

Class D.—There shall be two samples galvanized by the hot-dip process.

3. Basic commercial open-hearth iron. The analysis of this base material shall be:

Carbon, max	0.06 per cent
Manganese, max	0.15 "
Silicon	trace
Phosphorus, max	0.04 "
Sulfur, max	0.04 "
Copper, max	0.05 "

Samples.—The following samples are to be erected on each rack:

Class A.—None.

Class B.—None.

Class C.—There shall be two samples galvanized by the hot-dip process.

Class D.—There shall be two samples galvanized by the hot-dip process.

CHAIN LINK FENCE

The chain link fence shall be the manufacturer's standard chain link fence 42 in. high made of No. 9 gage wire galvanized after weaving. There are also to be exposed samples of manufacturer's standard chain link fence 42 in. high made of No. 9 gage wire galvanized before weaving.

Weight of Zinc Coating.—There shall be two weights of coating:

Class A.—Carrying a minimum weight of coating of 1 oz. of zinc per sq. ft. of surface.

Class B.—Carrying a maximum weight of coating of 0.4 oz. of zinc per sq. ft. of surface.

Zinc.—The zinc or spelter used for galvanizing shall conform in all respects to the requirements drawn up by Sub-Committee VI on Specifications for Metallic-Coated Products of Committee A-5.

Base Material.—1. Basic open-hearth steel without added copper. The analysis of this material shall be:

Carbon	0.08 to 0.15 per cent
Manganese, max	0.60 "
Silicon, max	0.12 "
Sulfur, max	0.06 "
Phosphorus, max	0.10 "
Copper, max	0.08 "

Samples.—The following samples are to be erected on each rack:

Class A.—There shall be two samples of the fence galvanized by the hot-dip process after weaving.

Class D.—There shall be two samples of the fence made from wire galvanized before weaving.

2. Basic open-hearth steel with added copper. The analysis of this base material shall be:

Carbon	0.08 to 0.15	per cent
Manganese, max	0.60	"
Silicon, max	0.12	"
Sulfur, max	0.06	"
Phosphorus, max	0.10	"
Copper, min	0.20	"

Samples.—The following samples are to be erected on each rack:

Class A.—There shall be two samples of the fence galvanized by the hot-dip process after weaving, and one galvanized by the hot-dip process with subsequent heat treatment before weaving (Keystone).

Class D.—There shall be two samples of the fence made from wire galvanized before weaving.

3. Basic commercial open-hearth iron. The analysis of this base material shall be:

Carbon, max	0.06	per cent
Manganese, max	0.15	"
Silicon	trace	
Phosphorus, max	0.04	"
Sulfur, max	0.04	"
Copper, max	0.05	"

Samples.—The following samples are to be erected on each rack:

Class A.—There shall be two samples of the fence galvanized by the hot-dip process after weaving.

Class D.—There shall be two samples of the fence made from wire galvanized before weaving.

STRUCTURAL SHAPES, HARDWARE, LINE MATERIALS, ETC.

TUBES

1. There shall be exposed at each location three sections 18 in. in length of 1-in. conduit of standard commercial base metal, the base material to be procured from the same source of supply, and with each of the following coatings: sherardizing, hot galvanizing, electrogalvanizing, calorizing, aluminizing, leadizing, chromium plating, and cadmium plating.

2. There shall be exposed at each location three sections 18 in. in length of 1-in. lap-welded pipe of standard commercial base metal, the base material to be procured from the same source of supply, and with each of the following coatings: sherardizing, hot galvanizing, electrogalvanizing, calorizing, aluminizing, leadizing, chromium plating, and cadmium plating.

BOLTS

There shall be exposed at each location three samples each of the following sizes of commercial square head bolts: ⅜ by 1½ in., ½ by 2½ in., ⅝ by 2½ in. and 1¼ by 6 in. in both plain and copper-bearing steel, with each of the following coatings, from two different producers of coatings where possible: hot-dipped galvanizing, electrogalvanizing, sprayed zinc, sherardizing in electric-heated drums, sherardizing in gas-heated drums, calorizing, aluminizing, chromium plating and cadmium plating.

HARDWARE AND CASTINGS

There shall be exposed at each location three samples each of the following articles: Malleable-iron suspension clamp, catalog No. 3122, Locke Insulator Corporation; malleable-iron line material clamp, cast-iron suspension clamp, catalog No. 3122, Locke Insulator Corporation; forging steel tower fitting, catalog No. 8435, Locke Insulator Corporation; cold-rolled steel standard form door hinge, with each of the following coatings: hot-dipped galvanizing, electrogalvanizing, sprayed zinc, sherardizing in electric-heated drums, sherardizing in gas-heated drums, calorizing, aluminizing, chromium plating and cadmium plating.

There shall be exposed at each location sufficient standard large-head 1-in. roofing nails, driven into the wooden cross arms, to permit of 5 being withdrawn at each semi-annual inspection for a period of twenty years, with each of the following coatings: hot-dipped galvanizing, electrogalvanizing, and sherardizing.

STRUCTURAL SHAPES

Each of the samples of structural shapes shall be supported at each end, being attached by means of porcelain tubes, ⅝ in. in diameter, porcelain washers with capscrews and nuts, in a manner hereinafter described.

Flats.—There shall be exposed at each location three samples of the following sizes of flat shapes: namely, 2½ in. by No. 14 gage, 3 by 1¼ in. in both plain and copper-bearing base metal conforming to the Society's Standard Specifications for Structural Steel for Buildings (A 9 – 24),[1] the base metal to be procured from the same source of supply, and with the following coatings: hot galvanizing, electrogalvanizing, sprayed zinc, sherardizing in electric-heated drums, sherardizing in gas-heated drums, calorizing, aluminizing, chromium plating and cadmium plating.

Angles.—There shall be exposed at each location three samples 18 in. in length of angles of the following sizes: 1½ by 1½ by ⅛ in., 2 by 2 by ⅛ in., and 3 by 3 by ¼ in., in both plain and copper-bearing steel conforming to the Society's Standard Specifications for Structural Steel for Buildings (A 9 – 24),[1] the base metal to be procured from the same source of supply, and with the following coatings: hot galvanizing, electrogalvanizing, sprayed zinc, sherardizing in electric-heated drums, sherardizing in gas-heated drums, calorizing, aluminizing, chromium plating and cadmium plating.

T-Bars.—There shall be exposed at each location three samples 18 in. in length of standard T-Bars 2¾ by 1¾ in. in both plain and copper-bearing steel conforming to the Society's Standard Specifications for Structural Steel for Buildings (A 9 – 24),[1] the base material to be secured from the same source of supply, and with the following coatings: hot galvanizing, electrogalvanizing, sprayed zinc, sherardizing in electric-heated drums, sherardizing in gas-heated drums, calorizing, aluminizing, chromium plating and cadmium plating.

Window Frame Sections.—There shall be exposed at each location three samples 18 in. in length of each of the following frame sections: standard window frame section, Catalog No. 161 of Detroit Steel Products Co., and standard window frame section, catalog No. 792, Detroit Steel Products Co., with each of the following coatings: hot-dipped galvanizing, electrogalvanizing, calorizing, aluminizing, sprayed zinc, chromium plating and cadmium plating.

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[1] **1924 Book of A.S.T.M. Standards.**

Respectfully submitted on behalf of the sub-committee,

F. A. HULL,
Chairman.

REPORT OF COMMITTEE A-7
ON
MALLEABLE CASTINGS

A joint meeting of Committee A-7 with the Committee on Malleable Iron of the American Foundrymen's Association was held in Buffalo on May 6. The committee recommends the following revision of the Standard Specifications for Malleable Castings (A 47 – 24).[1]

Section 3.—Add to the table of requirements a requirement on minimum yield point, as follows:

"Yield point, lb. per sq. in., minimum............ 30 000."

Add a new paragraph (*b*) to read as follows:

"(*b*) The yield point shall be determined by the drop of the beam of the testing machine."

The main object for the suggested revision has been occasioned by the fact that many engineers were uncertain as to the figure that safely might be used for this property in their calculations. This revision has been submitted to letter ballot of the committee with the following results: Affirmative 20, negative 1, not voting 4.

Some of the non-producers on the committee expressed a desire that additional specifications be prepared covering a grade of malleable iron of a lower quality than that covered by the present specifications, in the thought that a cheaper and more easily machineable product could be made. This question has been referred to a sub-committee of three for further consideration and report.

The committee reports with keen regret the death of Mr. H. R. Kimmel, who had been a valued member of this committee for some years.

This report has been submitted to letter ballot of the committee, which consists of 25 members, of whom 20 have voted affirmatively, one negatively, and 4 have refrained from voting.

Respectfully submitted on behalf of the committee,

W. P. Putnam,
Chairman.

Enrique Touceda,
Secretary.

Editorial Note

The proposed revisions of the Standard Specifications for Malleable Castings were accepted for publication as tentative and appear on page 897.

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[1] 1924 Book of A.S.T.M. Standards.

DISCUSSION

[Mr. Touceda presented on behalf of Committee A-7 the recommendation that a yield point requirement be added to the Standard Specifications for Malleable Castings (Serial Designation: A 47 - 24). —Ed.]

Mr. Gilbert.

Mr. H. W. Gilbert.[1]—The National Malleable and Steel Castings Co. are favorable toward any work that this Society has in hand that in its opinion will improve specifications or the material produced. When this proposal to add a yield point requirement first came to our attention, the National Company made a careful study of the specifications and the possible effect of adding to the physical properties the determination of the yield point by the drop of the beam method and also by the divider method. Owing to the limited time, we were unable to make as thorough an investigation as we would like to have made and as should be made before reaching a conclusion. Forty-eight tests on specimens selected equally from our six plants were carried out by our Research Department and witnessed by several of our engineers. The results of these tests showed that we were able to determine the yield point by the drop of the beam in only twelve cases, and we were able to determine the yield point by the divider method in all tests. It is our opinion, therefore, that to include the determination of the yield point by the drop-of-the-beam method in the specifications covering malleable castings will not prove satisfactory. We believe that this step of including the yield point is in the right direction but doubt very much that the recommended method of determining it is its proper one. From our point of view the graph representing the elongation of malleable iron begins as a straight line and continues so until the proportional limit is reached, and then breaks off into a gradual curve. So far as we have been able to determine, there is no perceptible dip in this graph. If this is correct, and we believe it is, we do not understand how it can be possible to determine the yield point of a malleable iron test specimen by the drop-of-the-beam method. It is, therefore, our opinion and we recommend that this subject be referred back to Committee A-7 for further investigation to make certain whether the yield point can be determined by the drop-of-the-beam method.

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[1] Manager, Department of Inspection and Tests, National Malleable and Steel Castings Co., Cleveland.

Mr. Gibboney. MR. J. H. GIBBONEY.[1]—May I ask Mr. Gilbert what was the elongation found in his tests?

Mr. Gilbert. MR. GILBERT.—The elongation would average about 15 or 16 per cent. I should say the range was probably from 12 to 20 per cent.

Mr. Gibboney. MR. GIBBONEY.—I am somewhat surprised at his findings in view of such high elongation results. I should judge that with the malleable iron produced some eight or ten years ago, there would be considerable difficulty in catching the yield point by the drop of the beam, but I am rather surprised at his not finding it with malleable iron showing 16 per cent elongation.

Mr. Gilbert. MR. GILBERT.—We account for it by the fact that we have been unable to find a dip in the elongation graph. In the case of the testing of a steel specimen, when the proportional or elastic limit is passed, there appears a very perceptible dip in the stress-strain graph and it is almost impossible to operate the testing machine past that point without the beam dropping. In the case of the graph representing a malleable test, there is no corresponding dip in the graph and we, therefore, do not see how it is possible to get a drop of the beam.

[The revision of the Standard Specifications was then accepted for publication as tentative.—ED.]

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[1] Chief Chemist, Norfolk and Western Railway Co., Roanoke, Va.

REPORT OF COMMITTEE A-9
ON
FERRO-ALLOYS

During the past year, Committee A-9 has held three meetings at which the reports of sub-committees were presented covering the work on specifications, methods of analysis and methods of sampling. Mr. F. M. Becket was elected vice-chairman of the committee and Mr. G. E. F. Lundell appointed as representative to Committee E-1 on Methods of Testing, and Mr. J. J. Crowe to Committee E-8 on Nomenclature and Definitions.

The Sub-Committee on Specifications submitted tentative specifications for spiegeleisen, tungsten powder, ferro-chromium, ferro-silicon, ferro-manganese and ferro-vanadium. The Sub-Committee on Analysis submitted tentative methods of analysis for ferro-silicon, ferro-manganese, ferro-chromium, ferro-vanadium, ferro-tungsten and tungsten metal, and the Sub-Committee on Sampling submitted tentative methods for sampling ferro-alloys; all of which are recommended for publication as tentative as appended hereto.[1]

In considering the specifications for ferro-alloys and methods of analysis and sampling, special consideration has been given to the coordination of the requirements of both the consumer and the manufacturer. Previously the specifications for ferro-alloys, as well as methods of analysis and sampling, were matters of independent agreement between the purchaser and seller. The first codifications of these standards was done by the Ordnance Department, U. S. A., and subsequently approved and adopted by the Federal Specifications Board and this work was taken as a foundation for the Committee's specifications. It will be noted that for the most part the proposed specifications are identical with those of the Federal Specifications Board, the only changes being to better define the product. The specifications are brief, but, in conjunction with the methods of analysis and sampling, it is considered that they are sufficiently broad to furnish an adequate basis of agreement between consumer and manufacturer in the majority of cases.

The committee has also discussed the question of the importance of tin and arsenic in high-speed steels. This is a matter that has occupied the attention of metallurgists for some time and seemed to be of sufficient importance to recommend to the Society that a special research committee be formed to investigate this matter. This committee has now been authorized and Mr. N. B. Hoffman appointed as chairman.

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[1] See pp. 528–533 and 537–567.—Ed.

Two new members have been elected to Committee A-9: the Crucible Steel Co. of America and the New Jersey Zinc Co., and it has also been credited with having secured two new memberships to the Society at large.

The proposed tentative standards submitted with this report were referred to letter ballot of the committee with the following results:

Items	Affirmative	Negative	Not Voting
1. Tentative Specifications for Tungsten Powder	12	1	2
2. Tentative Specifications for Spiegeleisen	12	1	2
3. Tentative Specifications for Ferro-Silicon	12	1	2
4. Tentative Specifications for Ferro-Chromium	12	1	2
5. Tentative Specifications for Ferro-Manganese	12	1	2
6. Tentative Specifications for Ferro-Vanadium	12	1	2
7. Tentative Methods of Sampling Ferro-Alloys	12	1	2
8. Tentative Methods of Chemical Analysis of Ferro-Alloys	13	2	0

This report has been submitted to letter ballot of the committee, which consists of 25 members, of whom 17 have voted affirmatively, none negatively, and 8 have refrained from voting.

Respectfully submitted on behalf of the committee,

F. C. LANGENBERG,
Chairman.

CHARLES MCKNIGHT, JR.,
Secretary

EDITORIAL NOTE

The Proposed Tentative Specifications for Tungsten Powder; for Spiegeleisen; for Ferro-Silicon; for Ferro-Chromium; for Ferro-Manganese; for Ferro-Vanadium; and the proposed Tentative Methods of Sampling Ferro-Alloys and the proposed Tentative Methods of Chemical Analysis of Ferro-Alloys were accepted for publication as tentative and appear on pages 528–533 and 537–567.

DISCUSSION

SPECIFICATIONS FOR SPIEGELEISEN

[Mr. Hoffman presented on behalf of Committee A-9 Proposed Tentative Specifications for Spiegeleisen.—ED.]

Mr. MacKenzie.

MR. J. T. MACKENZIE.[1]—We would like to see in the Specifications for Spiegeleisen some mention of the limit to which individual portions of a car can go. I remember not so long ago we got a car of spiegeleisen that averaged 20 per cent manganese, but after getting some very erratic results with it, we found out that there was some 9-per-cent material in the car, with enough 30-per-cent material to make the average. It seems to me that some mention of the uniformity of the material should be made in these specifications. Where the spiegeleisen is melted and put into a mixer, as in a good many steel plants, these variations make no difference, but where it is charged, as in iron foundries, in 50-lb. chunks, it makes considerable difference.

Mr. Hoffman.

MR. N. B. HOFFMAN[2] (*Chairman, Sub-Committee on Specifications of Committee A-9*).—Do you have any suggestions to offer as to a permissible variation in each shipment?

Mr. MacKenzie.

MR. MACKENZIE.—My suggestion would be that a 10 per cent allowance be made on not over 10 per cent of the shipment. Thus, in a 50-ton shipment of grade A spiegeleisen, no pig should be below 17 per cent manganese, and only 5 tons might be below 19 per cent, regardless of the average manganese content of the car. The maxima will be cared for automatically.

Mr. Hoffman.

MR. HOFFMAN.—That is a very good thought, and we will take it up in the committee.

[The specifications were then accepted for publication as tentative.—ED.]

SPECIFICATIONS FOR FERRO-SILICON

[Mr. Hoffman then presented on behalf of Committee A-9 Proposed Tentative Specifications for Ferro-Silicon.—ED.]

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[1] Chief Chemist, American Cast Iron Pipe Co., Birmingham, Ala.

[2] Chemist and Metallurgist, Colonial Steel Co., Pittsburgh, Pa.

Mr. Spring. MR. L. W. SPRING[1].—In the Specifications for Ferro-Silicon it is stated, "The ferro-silicon shall be furnished in lumps," if specified. Is there anything which requires the producer to furnish lumps which will not disintegrate? As a matter of fact, a great deal of lump ferro-silicon does disintegrate. Could that be covered in any way?

Mr. Hoffman. MR. HOFFMAN.—I think there is a very general understanding in the trade that ferro-silicon does disintegrate. The idea was to include a requirement whereby the ferro-silicon should not be all shipped as fines.

The Chairman. THE CHAIRMAN (*Past-President C. D. Young*).[2]—May I ask, Mr. Spring, if Section 2 (*b*), which reads, "When furnished in lump or granulated form the ferro-silicon shall be free from excessive disintegration," does not cover your point?

Mr. Spring. MR. SPRING.—It does not seem to me that it covers it. My idea in suggesting this is that if the committee has not considered the matter of disintegration at considerable length, it would be well for them to do so, because many times ferro-silicon is shipped which will be received in lump form, which after a short period of time will disintegrate spontaneously, and that is quite closely connected, so far as we have been able to find out, with the composition. The higher the silicon within this range of Grade A, the more it is liable to disintegrate. In other words, ferro-silicon over 50 per cent, especially over 52 per cent or around 52 per cent, is much more liable to disintegrate than that which has silicon 47 to 50 per cent. At one time, manufacturers suggested that phosphorus content had an influence and only recently a metallurgist of one manufacturer, in speaking of ferro-silicon disintegration, stated that they kept their phosphorus low to avoid it.

Some companies now are quite successful in furnishing lump ferro-silicon which does not disintegrate. Some companies apparently have not done it yet. With ferro-silicon over 50 per cent the disintegration is sometimes quite severe; between 47 and 50 per cent it will be very much less severe.

Mr. Hoffman. MR. HOFFMAN.—How long a time do they guarantee it to be stable?

Mr. Spring. MR. SPRING.—They do not guarantee it at present, but there is considerable competition and some manufacturers are quite successful in making ferro-silicon which does not disintegrate. We are receiving quite regularly carloads which are practically free from fines and remain so in stock for six months at least and apparently would stay so indefinitely.

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[1] Chief Chemist and Metallurgist, Crane Co., Chicago.

[2] Stores Manager, Pennsylvania Railroad System, Philadelphia.

MR. HOFFMAN.—We are certainly glad to have your valuable comments and the committee will endeavor to incorporate a paragraph covering your point. It may be a hardship, however, for most manufacturers to meet such requirements. **Mr. Hoffman.**

THE CHAIRMAN.—The Chair rules that this matter will be considered by the committee during the coming year, and a vote for the adoption of the tentative standard will include such consideration by the committee. **The Chairman.**

[The specifications were then accepted for publication as tentative.—ED.]

REPORT OF COMMITTEE B-1
ON
COPPER WIRE

In 1923 substantial agreement upon specifications for hard-drawn copper trolley wire with the American Electric Railway Association was reported and tentative specifications for trolley wire were presented to the Society and accepted for publication. It was explained at that time that these specifications, while jointly accepted by the American Electric Railway Engineering Association and the American Society for Testing Materials, were tentative and subject to further consideration. It is particularly pleasing to be able to report that final and complete agreement between the two societies on specifications for hard-drawn copper trolley wire has now been reached and appended hereto[1] is a revised draft of the tentative specifications for hard-drawn copper trolley wire, round and grooved, which are identical with those presented to and accepted by the Power Committee of the American Electric Railway Engineering Association. These specifications will be offered to that association at its annual meeting in the fall with the confident expectation that they will be accepted and approved. Therefore, Committee B-1 offers these specifications to our Society with the recommendation that they supersede the tentative specifications proposed in 1923[2] and that they be approved by the Society as standard.

The revised draft appended hereto[1] differs from the tentative specifications submitted in 1923 principally in wording and arrangement. The actual test requirements are practically unchanged. Both societies are to be congratulated upon having finally brought about complete agreement upon specifications for so important a product as trolley wire, and it may confidently be predicted that specifications so agreed upon will in time become the standard of the industry.

In the 1923 report, it was stated that the same joint committee was at work upon specifications for high-strength bronze trolley wire to supersede the present Standard Specifications for High-Strength Bronze Trolley Wire, Round and Grooved, 40 and 65-per-cent Conductivity (B 9 - 16).[3] This work has proceeded to a stage where tentative specifications have been agreed upon in all points save the matter of the twist test. It is agreed that the twist test should be qualitative rather than quantitative but that this wire should twist a certain number of times before breaking. Similar requirements are included in the hard copper trolley wire specifications and now the only difference in opinion between the representatives of the two societies is the proper number to designate as the minimum. It has been agreed to report the specifications to the two societies with the recommendation that they be accepted for publication as tentative. They will be identical except that each society will have its own minimum twist test. It is confidently to be expected that as a result of the experience gained after publication of the two specifications, proper ground for agreement will be found. The proposed tentative specifications are appended hereto[1] and are offered for publication as tentative.

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[1] See A.S.T.M. Standards Adopted in 1925.—Ed.

[2] Tentative Specifications for Round and Grooved Hard-Drawn Copper Trolley Wire (B 47 - 23 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 577; also 1924 Book of A.S.T.M. Tentative Standards, p. 66.

[3] 1924 Book of A.S.T.M. Standards.

Committee B-1 finds that the present Standard Specifications for Soft or Annealed Copper Wire (B 3 - 15) differ in form from the standard form of the Society. There has been no reason for technical revision of these specifications and they were recently offered to the General Sectional Committee on Insulated Wires and Cables organized under the Rules of Procedure of the American Engineering Standards Committee and they were finally recommended to that committee for formal approval by them. Action is still pending but there is no reason to anticipate failure of approval since each of the technical sub-committees concerned gave consideration to the specifications and recommended their acceptance without amendment. Committee B-1 has not heretofore undertaken a revision of the form of these specifications because it was felt that it would be desirable to postpone this until some technical revisions become necessary. Now that it has developed that no technical revision appears to be necessary in the near future, Committee B-1 has undertaken the revision of these specifications in form and arrangement without any change in substance and proposes to have any further editions of the specifications which may be printed appear in the revised standard form without making necessary any change in the date of approval.

The Standard Specifications for Hard-Drawn Wire (B 1 - 23) and for Medium-Hard-Drawn Wire (B 2 - 15) were offered for approval by the American Engineering Standards Committee and objection having been raised on certain technical points the specifications will undergo a revision by a sectional committee organized under the Rules of Procedure of the A.E.S.C. These specifications, if and when revised, will be reported back to this Society but until revised the existing specifications will continue as the standard of the American Society for Testing Materials.

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[1] See p. 578.—ED.

This report has been referred to letter ballot of the committee, which consists of 12 members, of whom 11 have voted affirmatively, none negatively, and 1 has refrained from voting.

Respectfully submitted on behalf of the committee,

J. A. CAPP,
Chairman.

EDITORIAL NOTE

The Tentative Specifications for Round and Grooved Hard-Drawn Copper Trolley Wire, amended as recommended in the report, were approved at the annual meeting and subsequently adopted by letter ballot of the Society on August 28, 1925, and appear in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

The proposed revisions of the Standard Specifications for High-Strength Bronze Trolley Wire, Round and Grooved, 40 and 65-per-cent Conductivity, were accepted for publication as new tentative specifications and appear on page 578.

REPORT OF COMMITTEE B-2
ON
NON-FERROUS METALS AND ALLOYS

During the past year the committee has been giving consideration to revisions in certain of its specifications and is recommending revisions in the following ones:

Lake and Electrolytic Copper Wire Bars;
Seamless Condenser Tubes;
Sheet Brass;
Brass and Copper Pipe;
Methods of Analysis for Aluminum;
Aluminum Base Alloy Sand Castings;
Aluminum Ingots.

New tentative specifications are proposed for Muntz metal condenser tube plates. The committee is recommending the advancement to standard of the Specifications for Seamless 70–30 Brass Condenser Tubes and Ferrule Stock and Seamless Muntz Metal Condenser Tubes and Ferrule Stock.

Revisions are being considered on certain other specifications, notably those for sheet aluminum and new specifications are being developed for certain high-strength light aluminum casting alloys, and for aluminum-base casting alloys in ingot form. These, it is hoped, will be in shape for presentation to the Society next year. A report prepared by H. K. Masters on the present status of the antimony situation is appended.

The details of the work of the committee can best be given by reference to the reports of the various sub-committees as presented at the last meeting of the committee, held March 20.

SUB-COMMITTEE I ON PURE METALS IN INGOT FORM

The main work of the sub-committee has been the standardization of the shapes and sizes of wire bars and the following revisions are offered for publication as tentative of the Standard Specifications B 4 – 13 and B 5 – 13.

Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 – 13).[1]

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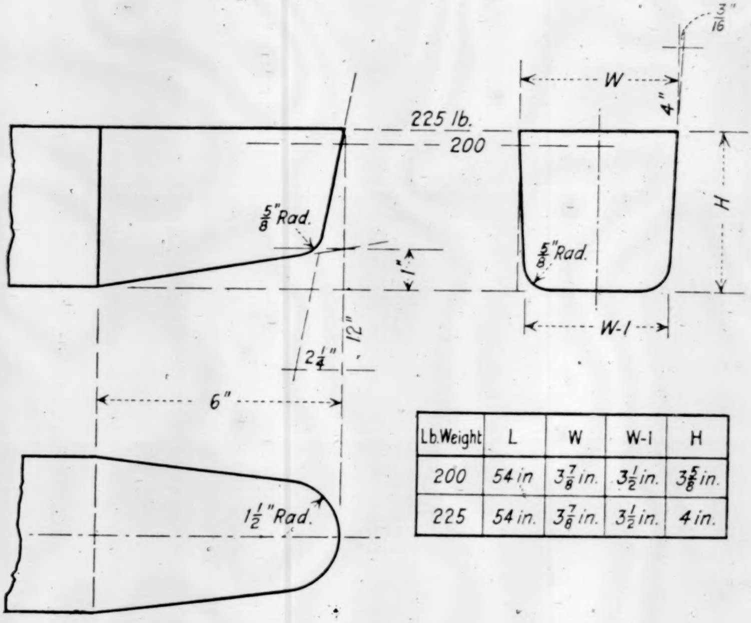
[1] 1924 Book of A.S.T.M. Standards.

Section 7.—Add a new Section 7 to read as follows, renumbering the subsequent sections accordingly:

"7. *Standard Sizes and Shapes for 200 to 300-lb. Wire Bars.*—(*a*) One mold shall be used for casting 200 to 230-lb. wire bars, the bottom width of this bar to be 3½ in., the listed weights being 200 and 225 lb. (See Fig. 1.)

"(*b*) One mold shall be used for casting 240 to 300-lb. wire bars, the bottom width of this bar to be 4 in., the listed weights being 250, 275 and 300 lb. (See Fig. 2.)



Lb. Weight	L	W	W-1	H
200	54 in.	3⅞ in.	3½ in.	3⅝ in.
225	54 in.	3⅞ in.	3½ in.	4 in.

FIG. 1

"(*c*) The length dimension of all bars shall be 54 in. The side draft or taper shall be ⅜ in. in 4 in. ($\frac{3}{16}$ in. in 4 in. on each side of the bar). The radius of the corners at the bottom of the bars shall be ⅝ in. The end taper at the bottom shall be 6 in. long overall and approximately 2 in. per ft.

"The end taper of the side shall be approximately 2¼ in. per ft. and the end of the bar shall be approximately 3⅜ in. deep at the point."

Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars (B 5 - 13).[1]

Section 6.—Add a new Section 6 to agree with the new Section 7 added to Specifications B 4 - 13 above, renumbering the subsequent sections accordingly.

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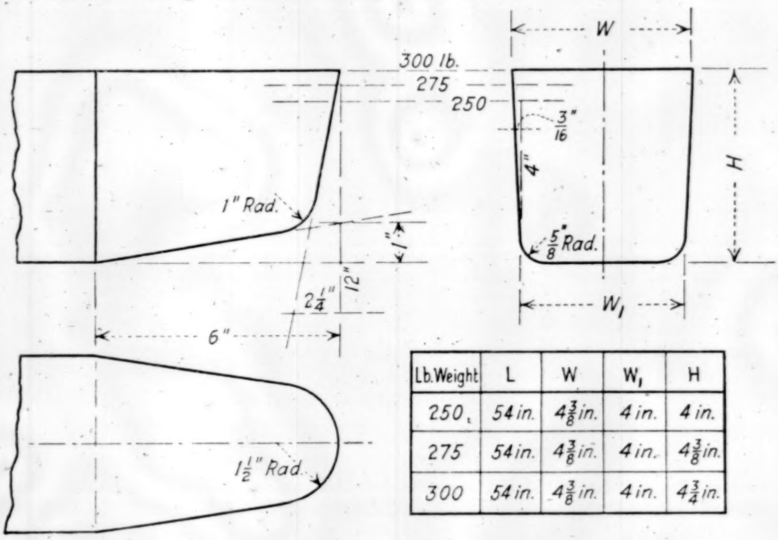
[1] 1924 Book of A.S.T.M. Standards.

Purchasers report the impossibility of securing pig lead according to the Society's Specifications B 29 – 23. The producers were represented on the committee drawing up these specifications and their representatives agreed to its provisions. The producers are reported as being unwilling to sell to the specifications, preferring to furnish material according to brand.



Lb. Weight	L	W	W_1	H
250	54 in.	$4\frac{3}{8}$ in.	4 in.	4 in.
275	54 in.	$4\frac{3}{8}$ in.	4 in.	$4\frac{3}{8}$ in.
300	54 in.	$4\frac{3}{8}$ in.	4 in.	$4\frac{3}{4}$ in.

FIG. 2.

SUB-COMMITTEE II ON WROUGHT NON-FERROUS METALS

The sub-committee recommends an amendment of the Standard Specifications for Seamless Admiralty Condenser Tubes and Ferrule Stock (B 44 – 24)[1] for publication as tentative.

Section 12.—Insert under Sub-Heading IV on Permissible Variations in Dimensions, a new Section 12 to read as follows, renumbering the subsequent sections accordingly:

12. *Diameter.*—The outside diameter of the tubes shall not vary from that specified by more than the following amounts, as measured by "go" and "no go" ring gages:

NOMINAL DIAMETER, IN.	PERMISSIBLE VARIATION, IN.
Up to 0.500, inclusive	plus or minus 0.002
Over 0.500 to 0.740, inclusive	" " " 0.0025
Over 0.740 to 1.000, "	" " " 0.003
Over 1.000 to 1.250, "	" " " 0.0035
Over 1.250 to 1.500, "	" " " 0.004

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[1] 1924 Book of A.S.T.M. Standards.

No criticisms have been received within the year as to the Tentative Specifications for Seamless 70-30 Brass Condenser Tubes and Ferrule Stock (B 55 – 24 T),[1] and Seamless Muntz Metal Condenser Tubes and Ferrule Stock (B 56 – 24 T),[1] and it is recommended that these be advanced to standard.

The sub-committee was requested to prepare specifications covering Muntz Metal Condenser Tube Plates. These specifications have been prepared and are submitted herewith[2] with the recommendation that they be accepted as a tentative standard.

TABLE I.—PERMISSIBLE VARIATIONS IN THICKNESS OF HIGH SHEET BRASS, IN.

Thickness		Width, in			
B. & S. Gage No.	Inch	Up to 6 in., inclusive	Over 6 to 9 in., inclusive	Over 9 to 14 in., inclusive	Over 14 to 20 in., inclusive
0000	0.460	0.0043	0.0045	0.0047	0.0049
000	0.4096	0.0042	0.0044	0.0046	0.0048
00	0.3648	0.0041	0.0043	0.0045	0.0047
0	0.3249	0.0040	0.0042	0.0044	0.0046
1	0.2893	0.0039	0.0041	0.0043	0.0045
2	0.2576	0.0038	0.0040	0.0042	0.0044
3	0.2294	0.0037	0.0039	0.0041	0.0043
4	0.2043	0.0036	0.0038	0.0040	0.0042
5	0.1819	0.0035	0.0037	0.0039	0.0041
6	0.1620	0.0034	0.0036	0.0038	0.0040
7	0.1443	0.0033	0.0035	0.0037	0.0039
8	0.1285	0.0032	0.0034	0.0036	0.0038
9	0.1144	0.0031	0.0033	0.0035	0.0037
10	0.1019	0.0030	0.0032	0.0034	0.0036
11	0.0907	0.0029	0.0031	0.0033	0.0035
12	0.0808	0.0028	0.0030	0.0032	0.0034
13	0.0719	0.0027	0.0029	0.0031	0.0033
14	0.0640	0.0026	0.0028	0.0030	0.0032
15	0.0570	0.0025	0.0027	0.0029	0.0031
16	0.0508	0.0024	0.0026	0.0028	0.0030
17	0.0452	0.0023	0.0025	0.0027	0.0029
18	0.0403	0.0022	0.0024	0.0026	0.0028
19	0.0359	0.0021	0.0023	0.0025	0.0026
20	0.0320	0.0020	0.0021	0.0023	0.0024
21	0.0284	0.0019	0.0020	0.0021	0.0022
22	0.0253	0.0018	0.0019	0.0020	0.0021
23	0.0225	0.0017	0.0018	0.0019	0.0020
24	0.0201	0.0016	0.0017	0.0018	0.0019
25	0.0179	0.0015	0.0016	0.0017	0.0018
26	0.0159	0.0014	0.0015	0.0016	0.0017
27	0.0142	0.0013	0.0014	0.0015	0.0016
28	0.0126	0.0012	0.0013	0.0014	0.0015
29	0.0112	0.0011	0.0012	0.0013	0.0014
30	0.0100	0.0011	0.0012	0.0013	0.0014
31	0.0089	0.0010	0.0011	0.0012	0.0013
32	0.0079	0.0010	0.0011	0.0012	0.0013
33	0.0070	0.0009	0.0010	0.0011	0.0012
34	0.0063	0.0009	0.0010	0.0011	0.0012
35	0.0056	0.0008	0.0009	0.0010	0.0011
36	0.0050	0.0008	0.0009	0.0010	0.0011

The sub-committee has given consideration to the matter of gage tolerances for sheet brass, and recommends the substitution of the accompanying Table I covering permissible variations in thickness for Table I in Section 13 of the Standard Specifications for High Sheet Brass (B 36 – 21).[3]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, pp. 794, 799 (1924); also 1924 Book of A.S.T.M. Tentative Standards, pp. 81, 86.

[2] See p. 583.

[3] 1924 Book of A.S.T.M. Standards.

SUB-COMMITTEE III ON SAND-CAST METALS AND ALLOYS.

Due to the illness of the chairman of the sub-committee, at the time of the March meeting of Committee B-2, it was not possible to present and give consideration at that time to the sub-committee's report. The sub-committee has, however, been active during the year and has developed four new specifications, namely, for aluminum-bronze castings, for composition brass ounce metal, for 88-8-4 bronze and for steam or valve bronze. It is hoped to present these specifications to the Society next year. At the same time certain revisions have been discussed and will be recommended next year in the Standard Specifications for Bronze Bearing Metals for Turntables and Movable Railroad Bridges (B 22 - 21) and the Standard Specifications for Bronze Bearing Metal in Ingot Form (B 31 - 21), with reference to changing the phosphorus content from a minimum of 0.75 to a minimum of 0.25 per cent.

The Standard Specifications for the Alloy: Copper, 88 per cent; Tin, 10 per cent; Zinc, 2 per cent (B 10 - 18) and the Tentative Specifications for Manganese Bronze Ingots for Sand Castings (B 7 - 24 T) have also been discussed in the sub-committee and recommendations will be made next year concerning certain minor revisions.

The work on standard test bars for cast non-ferrous metals is progressing and it is hoped that the report concerning it can be made to the Society at its 1926 meeting.

SUB-COMMITTEE IV ON WHITE METALS

The sub-committee reported that none of the research work, namely, studying the effects in the following respects of the added impurities to white metals:

1. Upon Wear, by conducting friction and wear tests;
2. Upon fluidity, by devising if possible a test for the determination of fluidity;
3. Upon drossing;
4. Upon brittleness;

had been undertaken during the year. This has been due to the fact that the Bureau of Standards has been unable to find time and appropriations for the continuance of this work. A communication from H. W. Gillett, chief, Division of Metallurgy, Bureau of Standards, stated the reasons why the work had not been undertaken and also stated that he did not think it would be possible to begin the work in the near future unless it could be shown that it was of sufficient importance.

Since the last meeting the values given in the table appended to the Tentative Specification for White Metal Bearing Alloys (B 23 - 18 T) were revised to conform to the determinations made by the Bureau, and in the revised form were printed in the 1924 Book of A.S.T.M. Tentative Standards.

SUB-COMMITTEE VI ON NON-FERROUS METALS FOR RAILROAD EQUIPMENT

The sub-committee reported that distinct progress has been made toward a unification of the specifications of the American Railway Association and of the American Society for Testing Materials. The chairman of the A.R.A. Committee on Specifications covering engineering materials used in railway service, has agreed to submit to the special A.R.A. Committee on Non-Ferrous Alloys for Railway Equipment, the recommendations of, also the criticisms of the A.R.A. specifications submitted by, Sub-Committee VI. Recommendations were agreed to regarding the composition of the backs of car journal bearings and also the lining metal.

It is the hope of the sub-committee that the A.R.A. committee, with these recommendations and criticisms before it, will see fit to modify its specification to such extent that it will be acceptable to Sub-Committee VI, and that the specifications of the two organizations can be made identical.

SUB-COMMITTEE VII ON METHODS OF CHEMICAL ANALYSIS

The work of Sub-Committee VII has principally been in connection with the revision of the Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (B 40 - 23 T)[1] based upon cooperative analytical work of several laboratories on samples of commercial aluminum and aluminum alloys furnished by the chairman of Sub-Committee VIII on Aluminum Alloys and the discussion which has followed this work. The following changes are recommended in these methods. The page references are to Part I of the Proceedings for 1923.

On Page 597.—Change sub-title A to read: "Determination of Silicon and Titanium."

In sub-heading in next line, change "Non-Graphitic" to "Total," making the sub-heading read: "Determination of Total Silicon."

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 597 (1923); also 1924 Book of A.S.T.M. Tentative Standards, p. 110.

On Page 598.—Change the sentence beginning on line 4 to read as follows, by the addition of the italicized word and the omission of those in brackets:

"Wash the residue with hot water until free from sulfates *and* ignite in a platinum crucible [and weigh]."

Omit the rest of the paragraph and insert the following:

"Fuse the residue with 8 to 10 times its weight of Na_2CO_3 and treat the fused mass in a casserole or beaker with 20 cc. of H_2SO_4 (1:3), washing out the crucible. Evaporate the solution until it fumes strongly to separate the SiO_2. Cool, dilute to about 100 cc., boil till the salts are dissolved, filter, wash well, ignite in a platinum crucible and weigh. Treat the ash with HF and a few drops of H_2SO_4 (1:3), carefully evaporate till dry, ignite and again weigh.

"From the difference between the two weights deduct a blank obtained from the reagents and filter. The rest of the loss in weight represents SiO_2 corresponding to the Si in the metal.

$$SiO_2 \times 0.4672 = Si."$$

Delete entirely the sub-headings and directions for "Determination of Graphitic Silicon" and "Determination of Total Silicon."

Under "Notes," retain the first note consisting of one line and delete entirely the second paragraph beginning with "Graphitic Silicon . ." and ending with ". . the best that has been devised."

Insert the following note:

"In routine analysis, fusion of the first H_2SO_4 residue with sodium carbonate and subsequent treatment to the point of treating the ash with HF may be omitted. The first residue may be weighed and treated with HF, etc., as directed for the second H_2SO_4 residue. This, however, should not be done if the material contains much graphitic silicon or if the residue after ignition is dark colored."

On page 599.—Under "Method," first line, change "Graphitic" to "total," making the line read: "See 'Determination of Total Silicon'."

In the first line of the next paragraph, delete the word "graphitic."

In the third line of same paragraph, make the word "filtrate" plural.

In the third and fourth lines from the end of the paragraph, delete the sentence:

"Reserve the tested solution for the determination of iron."

On page 600.—Insert after the sub-title "Determination of Iron" the following entire method, in place of the present method, deleting all of the matter on this page and three lines at top of page 601:

DETERMINATION OF IRON

SOLUTIONS REQUIRED

"'*Acid Mixture.*'—1200 cc. of H_2SO_4 (25-per-cent by volume), 600 cc. of HCl (sp. gr. 1.20) and 200 cc. of HNO_3 (sp. gr. 1.42).

"*Tartaric Acid Solution.*—Dissolve 20 g. of tartaric acid crystals in 100 cc. of distilled water.

"*Dilute Ammonium Sulfide Solution.*—Add 15 cc. of NH_4OH (sp. gr. 0.90) to 15 cc. of distilled water and saturate with H_2S; then dilute to 200 cc. with distilled water. Use freshly made.

"*Dilute Sulfuric Acid Solution for Reductor* (*5-per-cent*).—Mix 50 cc. of H_2SO_4 (sp. gr. 1.84) and 1000 cc. of distilled water.

"*Standard Potassium Permanganate Solution.*—Dissolve 1 g. $KMnO_4$ in 1000 cc. of H_2O, and allow to stand for several days in a glass-stoppered bottle in a dark closet. Filter the solution through prepared asbestos and standardize against 0.1000 g. portions of pure sodium oxalate. Each cubic centimeter is equivalent to approximately 0.00177 g. of iron.

METHOD

"Dissolve 1 g. of the well-mixed sample in 35 cc. of the acid mixture and proceed as in 'Determination of Silicon,' including treatment with HF omitting the weighing of residues. Fuse the last residue obtained with a small amount of $K_2S_2O_7$, take up in a little 5-per-cent H_2SO_4, and add to the filtrates and washings obtained. Saturate this solution with H_2S and filter. To the filtrate add 25 cc. of the tartaric acid solution, and then with constant stirring, add NH_4OH (sp. gr. 0.90), finally drop by drop, until it is in slight excess. Pass in H_2S for a few moments, warm somewhat and allow the solution to stand. Filter and wash the sulfide precipitate thoroughly with a freshly made dilute ammonium sulfide solution, until all tartaric acid is washed out.

"Dissolve the sulfide precipitate thorough the filter paper with warm dilute H_2SO_4 (1:10) washing the paper thoroughly, alternately with hot distilled water and with 5-per-cent H_2SO_4. Boil until all H_2S is expelled and add HNO_3 (sp. gr. 1.42) until the iron is oxidized, which is shown by the solution becoming of a yellowish color. Add 3 to 4 g. of NH_4Cl and then NH_4OH (sp. gr. 0.90) in excess until a permanent precipitate forms. Boil again, allow to settle and filter, washing with dilute ammonia and hot distilled water.

"Dissolve the washed precipitate through the filter with warm dilute H_2SO_4 (1:10), wash the paper thoroughly with 5-per-cent H_2SO_4 and hot distilled water, using in all about 100 cc. Pass the solution through the reductor and wash the beaker and reductor with 150 cc. of dilute H_2SO_4 for reductor and then 100 cc. of distilled water. Titrate with standard $KMnO_4$ solution.

NOTES

"Results of titration should be checked with a blank determination made on corresponding amounts of solutions used.

"A small quantity of liquid should always be left in the reductor funnel and air should never be allowed to enter the body of the reductor.

"For further details of the use of reductor see The Chemical Analysis of Iron by A. S. Blair, or Quantitive Chemical Analysis by H. P. Talbot.

"The effect of tartaric acid is to form complexes with aluminum, vanadium, chromium, titanium, etc., preventing their precipitation as hydroxides.

"Where the iron exists in very small quantities, a gravimetric method is preferable. In this case, double precipitation of iron sulfide is necessary to eliminate all aluminum. Dissolve the second sulfide precipitate in warm dilute H_2SO_4 (1:10), boil, and oxidize the iron with H_2O_2. Add 3 to 4 g. of NH_4Cl and then NH_4OH (sp. gr. 0.90) in considerable excess, with stirring. Filter quickly, dry and ignite the precipitate, weighing as ferric oxide. If an accurate determination of iron is desired, it is necessary to correct for the small accumulation of silica by treatment of the ignited precipitate with $H_2SO_4 \cdot HF$."

On Page 601.—Make the sub-title read "Determination of Copper by Electrolytic Method." Delete the letter "(*a*)" designating this method as one of two optional methods.

Under "Solutions Required" delete the entire two lines consisting of directions for mixing dilute sulfuric acid solution. Insert under "Solutions Required" the following:

"*Sodium Hydroxide Solution.*—Dissolve 250 g. of NaOH in distilled water and dilute to 1000 cc.

Nitric Acid (1:1)—Pour 500 cc. of HNO_3 (sp. gr. 1.42) into distilled water and dilute to 1000 cc."

Under "Method," change the first four sentences from their present form: namely,

"Dissolve 10 g. of the sample in 100 cc. of dilute H_2SO_4 (1:1). When action has ceased add 25 cc. of HNO_3 (sp. gr. 1.42) and boil until oxides of nitrogen have been expelled. Filter into the tall form of beaker used in electrolysis and wash the residue with hot water. Dilute to 400 cc. and electrolyze."

to read as follows:

"Dissolve 10 g. of the sample in a large casserole or beaker with 150 cc. of the NaOH solution. This should be added in small portion as the action is vigorous for a time. After the action slackens, complete solution may be hastened by warming. When no evidence of further reaction can be seen, dilute to about 700 cc. with water at the boiling temperature. Filter through a strong paper and wash.

"Dissolve the residue from the paper with 25 cc. of hot HNO_3 (1:1) into a beaker for electrolyzing, and wash the filter thoroughly with hot water. Boil to expel oxides of nitrogen, add 6 to 8 cc. of H_2SO_4 (1:1), dilute to about 150 cc. and electrolyze."

Under "Notes" at the bottom of the page in the second paragraph, second and third lines, delete "as in the optional method."

On page 602.—Omit the entire page comprising Iodide Method with directions for solutions required and description of method.

On page 603.—Include the note at the top of the page with the notes at the bottom of page 601.

In the "Determination of Manganese" under "Solutions Required" delete entirely the two lines giving directions for mixing dilute sulfuric acid. In place of this, insert the following:

"*Acid Mixture.*—Pour 400 cc. of H_2SO_4 (sp. gr. 1.84) into distilled water, cool and add 400 cc. of HNO_3 (sp. gr. 1.42). Then dilute the mixture with distilled water to a total volume of 2000 cc."

Change the first sentence of the paragraph under "Method" from its present form: namely,

"In a 250-cc. Erlenmeyer flask, dissolve 0.5 g. of the sample in 25 cc. of H_2SO_4 (1:1) by warming and adding a drop or two of HNO_3 (sp. gr. 1.42) occasionally until solution is complete."

to read as follows:

"In a 250-cc. Erlenmeyer flask, dissolve 1 g. of the sample in 30 cc. of acid mixture by warming on a water bath until solution is complete."

On page 604.—Insert under "Notes" an additional one as follows;

"If it is desired, larger amounts of the sample can be used and correspondingly greater amounts of $AgNO_3$, NaCl and $(NH_4)_2S_2O_8$ (preferably in the form of stronger solutions)."

On page 605.—Under "Notes" insert an additional one as follows:

"The sodium bismuthate reagent should be tested for manganese and used only if its absence is shown."

Omit entirely "Determination of Carbon" with the description of solutions required and method ending with five lines at the top of page 606.

On page 606.—In title *A*, delete words "Non-Graphitic, Graphitic and Total," making the title read: "Determination of Silicon, Titanium and Iron."

In the next sub-heading, insert the word "Total" making it read: "Determination of Total Silicon." Delete entirely the words in parentheses under this sub-heading: "Non-Graphitic, Graphitic and Total Silicon."

On page 607.—Under "Determination of Iron," delete the line reading: "Proceed as described under 'Determination of Iron in Aluminum'" and insert the entire method with solutions required, description of methods and notes, as it appears on page 600. This includes the whole of page 600 and three lines completing the notes at the top of page 601. In making this insertion, change the word "reduction" to "reductor" in the directions for dilute sulfuric acid under "Solutions Required," and omit the sentence reading as follows:

"This solution is used boiling hot."

Include with the four notes as given on page 600 the first two notes given on page 607. Delete the third note on this page reading:

> "For the determination of iron in the presence of titanium see Notes under 'Determination of Iron' in Aluminum."

On page 611.—Delete "Determination of Copper by the Iodide Method (Optional)" and directions for carrying out this method consisting of 5 lines.

On page 617.—Delete "Determination of Carbon" and the one line below referring to this method.

SUB-COMMITTEE VIII ON ALUMINUM ALLOYS

Sub-Committee VIII presents the following revisions of the Tentative Specifications for Light Aluminum Casting Alloys (B 26 - 24 T):[1]

Title.—Change to read as follows:

> "Aluminum Base Alloy Sand Castings."

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> "These specifications cover commercial [light alloys of] aluminum *base alloy sand castings* having a specific gravity of 3 and less."

Section 5.—Change the minimum tensile strength requirement for alloy D from 18,000 lb. per sq. in. to 19,000 lb. per sq. in.

Section 6.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> "(*a*) Two test bars [of the form and dimensions shown in Fig. 1] shall be an integral part of large castings, or cast separately in the case of small castings to represent a lot or melt, and shall be molded in a manner similar to the castings which they represent. If the castings are heat treated, the test bars representing such castings shall be similarly heat treated.
>
> "(*b*) The manufacturer and purchaser shall agree whether *the* test bars can be attached to castings, on the [location] *locations* of the bars [on the castings], on the castings to which bars are to be attached, [and] on the method of casting unattached bars *and whether a 'cast to size bar' or a 'machined to size bar' shall be used.*
>
> "(*c*) [Tension test specimens, turned from the test bar shown in Fig. 1, shall conform to the dimensions shown in Fig. 2.] *The 'machined to size bar' shall be cast according to the dimensions shown in Fig. 1 and machined to conform to the dimensions shown in Fig. 2. The 'cast to size' bar shall be cast according to the dimensions shown in Fig. 2 and tested without machining.* The ends shall be of a form to fit the holders of the testing machine in such a way that the load shall be axial."

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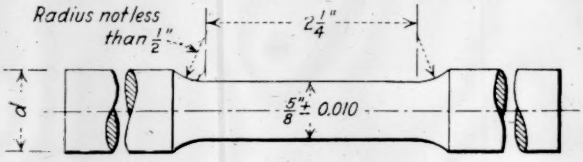
[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 785 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 62.

Figures 1 and 2.—Change Figs. 1 and 2 to conform to the accompanying Figs. 3 and 4, respectively.

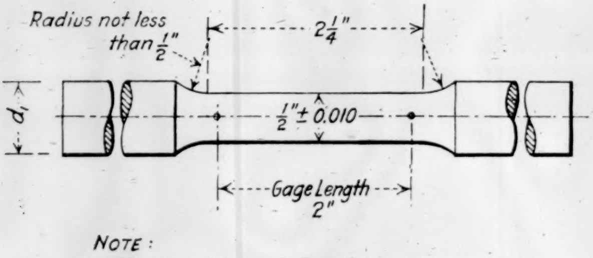
Explanatory Notes.—Omit the first sentence of the Explanatory Notes reading as follows:

"These alloys are all suitable for general sand casting purposes."



NOTE:
The Gage Length, Parallel Section and Fillets shall be as shown, but the Ends may be of any Shape to permit of Machining according to the Requirements of Fig. 4.
It is recommended, that Diameter d be from $\frac{7}{8}$" to $1\frac{1}{8}$".

FIG. 3.



NOTE:
The Gage Length, Parallel Section and Fillets shall be as shown, but the Ends may be of any Shape to fit the Holders of the Testing Machine in such a way, that the Load shall be Axial.
It is recommended, that Diameter d_1 be from $\frac{3}{4}$" to 1."

FIG. 4.

Change the first paragraph of the Explanatory Notes relating to Section 6 to read as follows:

"Some latitude has been allowed in the matter of test specimens; test bars 'cast to size' are stronger than those which are 'machined to size' and the tensile properties specified are regularly obtained in commercial production only with the 'cast to size' bar. When 'machined to size' specimens are required to meet the values given in these specifications, rejections may be expected to run higher than those experienced in usual commercial production. Owing to insufficient information being available on the tensile properties of 'machined to size' specimens it was not possible to include a separate table of values for this type of specimen."

Revisions of the Tentative Specifications for Aluminum Ingots for Remelting and for Rolling (B 24 - 22 T)[1] have also been considered and are recommended as follows:

Title.—Change the title to read:

"Aluminum Ingots for Remelting."

Section 3.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

"No scrap shall be used other than that resulting in the manufacturer's own plant from the production of material [complying with these specifications, and which is of similar composition to the material specified] *of composition similar to that specified.*"

Section 4.—Change from its present form: namely,

"Ingots for rolling shall be of uniform quality, tough and sound. They shall be substantially free from blowholes, shrinkage cracks, cold-sets, dross, foreign matter, and other defects in set or casting. Such defects may be waived in ingots which are to be remelted."

to read as follows:

"Aluminum ingots shall have a clean surface and shall be free from dirt, slag and foreign material."

Section 6.—Change the first sentence to read as follows by the addition of the italicized words and the omission of the word in brackets:

"The ingots selected shall be drilled *without lubricant* completely through or half *way* through from top and bottom."

Sections 9 and 10.—Change so as to agree in form with the usual sections on claims.

The sub-committee is engaged also in drawing up specifications for aluminum base casting alloys and for high-strength alloy castings which it is hoped will be ready for the next meeting.

Revisions are also under consideration for the Tentative Specifications for Aluminum Sheet (B 25 - 19 T).

SUB-COMMITTEE X ON METHODS OF TESTING

The sub-committee has made inquiry of the other sub-committees regarding the practice of the determination of yield point in tension on non-ferrous alloys and finds that the practice is universal of using as yield point, that stress which produces an extension of 0.5 per cent (under load). This practice is, however, not followed in the case of the testing of aluminum base alloys. The sub-committee is therefore recommending that except in the case of specifications for aluminum base alloys, the present specifications for other non-ferrous alloys in which tension tests are required specify the method of determination of yield point when the latter is mentioned. This will require only one amendment, namely, in the Standard Specifications for Naval Brass Rods for Structural Purposes (B 21 - 19)[1] as follows:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 646 (1922); also 1924 Book of A.S.T.M. Tentative Standards, p. 99.

Section 6.—Add a paragraph to read as follows:

> "(*b*) Yield point in tension shall be determined as the stress producing an elongation under load of 0.5 per cent, that is, 0.01 in. in a gage length of 2 in."

The analysis of the letter ballot of the committee on the recommendations contained in this report is as follows:

Item	Affirmative	Negative	Not Voting
I. PROPOSED REVISIONS IN STANDARDS			
1. For Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars (B 4 - 13)	32	0	68
2. For Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars (B 5 - 13)	31	0	69
3. For Seamless Admiralty Condenser Tubes and Ferrule Stock (B 44 - 24)	34	1	65
4. For High Sheet Brass (B 36 - 21)	30	0	70
5. Insertion of Method of Determination of Yield Point in Specifications for Naval Brass Rod for Structural Purposes (B 21 - 19)	32	1	67
II. PROPOSED REVISIONS IN TENTATIVE STANDARDS			
6. For Light Aluminum Casting Alloys (B 26 - 24 T)	30	0	70
7. For Aluminum Ingots for Remelting and for Rolling (B 24 - 22 T)	30	0	70
8. Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (B 40 - 23 T)	28	0	72
III. PROPOSED NEW TENTATIVE STANDARDS			
9. For Muntz Metal Condenser Tube Plates	30	0	70
IV. TENTATIVE STANDARDS ADVANCED TO STANDARD			
10. For Seamless 70-30 Brass Condenser Tubes and Ferrule Stock (B 55 - 24 T)	29	0	71
11. For Seamless Muntz Metal Condenser Tubes and Ferrule Stock (B 56 - 24 T)	29	0	71

This report has been submitted to letter ballot of the committee, which consists of 100 members, of whom 46 have voted affirmatively, none negatively and 54 have refrained from voting.

Respectfully submitted on behalf of the committee,

WILLIAM CAMPBELL,
Chairman.

P. D. MERICA,
Secretary.

For Addendum to this report, see page 149.—ED.

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[1] 1924 Book of A.S.T.M. Standards.

APPENDIX

REVIEW OF THE ANTIMONY SITUATION

By H. K. Masters[1]

The situation with respect to specifications for refined antimony is similar to that described in "A Review of the Tin Situation" by Lawrence Addicks.[2] Much the same conditions prevail with respect to the great geographical distance between the points of production of antimony metal and its consumption. The United States is dependent on foreign sources of supply. This is primarily due to the greater resources of antimony in certain foreign countries, particularly China, but is also largely on account of lower wage scales abroad. For the latter reason, our known antimony deposits remain mostly undeveloped and other deposits which may be greater than is generally assumed remain undiscovered. Of the entire world's production of antimony, 80 to 90 per cent comes from China; but with the lack of any well-organized smelting industry in that country, it is practically impossible to impose any specification on producers other than that the metal shall contain a minimum of 99 per cent antimony.

About all that the user of the metal can do is to require, first, a uniform grade; second, that the metal shall be as free from sulfur and arsenic as possible; and third, that its quality shall be such that when remelted very little dross is produced. As in the case of tin, the consumer buys the metal, guaranteed minimum 99 per cent antimony, specifying brand if possible, and then controls his operations by means of analyses in his own laboratory.

This review is submitted in place of a specification in order to give a basis for discussion and to present information on production and present commercial practice in marketing antimony that should be helpful to consumers.

Ores.—By far the most important commercial ore of antimony is stibnite, also known as antimonite, antimony glance and gray antimony. It is the sulfide Sb_2S_3, containing when pure 71.4 per cent of antimony. The mineral is commonly found as a confused aggregate of needle-shaped crystals.

When stibnite undergoes decomposition, the resulting alteration products are the less important oxidized ores: Valentinite, senarmontite, kermesite and cervantite.

Geographical Distribution.—China is the largest producer and from a time shortly after the outbreak of the World War has controlled the market. The most important and extensive deposits are in the Southern Provinces and apparently no antimony deposits occur north of the Yangste River. The Province of Hunan produces about five-sixths of the output of all China. The ore is hand-sorted or hand-washed, the former running about 60 per cent antimony and the latter about 45 per cent. The impurities are: iron, 0.5 per cent; arsenic, 0.10 per cent; zinc, 0.05 per cent; and traces of lead and copper. According to C. Y. Wang the ore reserves of this region are estimated at about 4,000,000 tons of 50-per-cent ore and the production over 1915 to 1921, inclusive, 200,000 tons of 55-per-cent ore.

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[1] Secretary, New York Metal Exchange, New York City.

[2] Lawrence Addicks, *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, page 184 (1923).

Prior to the War the principal producing countries besides China were France, Mexico, Austria-Hungary, Italy, Serbia, Algiers, Bolivia and Australia. Relatively less important producers have been Portugal, Spain, Borneo, Japan, Canada and the United States. Under the stimulus of war high prices, the United States produced 5000 tons of ore in 1915 and 4500 in 1916, from the following states in order of importance: California, Nevada, Washington, Idaho, Arkansas, Utah, Arizona and Montana, and also from the Territory of Alaska.

At a conference before the U. S. Tariff Commission on the Antimony Industry of the United States, held in San Francisco, June 24, 1918, the situation in this country was very carefully canvassed. Producers, consumers and importers were represented and the consensus of opinion seemed to be that the extreme price fluctuations of the antimony market made the development of any industry in this country very difficult. Apparently, a steady price of about 16 cents was regarded as necessary to stimulate antimony mining. One of the California properties then operating was reported as being very disappointing in its development while another gave evidence of large possibilities. Producers seemed to consider a tariff of 6 cents on the metal absolutely necessary in order to develop an industry able to compete with the Chinese. At that conference, developments in the electrolytic production of antimony were reported as disappointing but the possibilities of flotation as applied to low-grade ores were very strongly brought out.

Metallurgy.—Smelting processes may be divided broadly into four classes:

1. Rich ores and liquated sulfide are dead-roasted in hearth or reverberatory furnaces and the roasted ore melted with soda ash and carbon in small reverberatory furnaces to metal, or "regulus."
2. Rich sulfide ores are heated with little or no air. A large part of the sulfide melts and runs to the bottom of the furnace and is tapped off. This is the liquated sulfide mentioned above. The residues, plus lean ores mixed with coal, are smelted in shaft (Herrenschmidt) furnaces. The antimony is here driven off and is deposited in settling chambers and flues as an impure oxide. This is treated with roasted rich ore in the metal furnace.
3. Blast-furnace smelting of antimony ores was developed commercially in the United States during the War.
4. The English direct smelting process (also used to some extent in China during the war) eliminates the roasting expense. Only rich ores are smelted by this process, since the cost of the necessary fluxes prohibits the use of low-grade ore. The charge consists of ore, scrap iron (to take care of sulfur), salt and slag, which is melted in crucibles in a reverberatory furnace with condensing chambers. The product of the first melt is known as "single" metal, about 91 per cent antimony. This is melted with a small amount of crude (liquated sulfide) and salt to remove any iron left and then cast as "doubles" metal or "star bowls," which is later refined to "star" regulus, as follows: The "doubles" metal is melted in a crucible furnace with an alkaline flux or slag which refines the metal to 99.5 per cent antimony. This is cast into 30 to 40-lb. ingots, ready for the market. The alkaline flux used accomplishes two purposes: it refines the antimony in the furnace and permits it to solidify while the cover of flux is still fluid. This produces the "star" appearance considered by the trade as an indication of superior quality of this "regulus."

In referring to the smelting methods of the Chinese, C. Y. Wang in his article in the Transactions of the American Institute of Mining Engineers, September, 1918, stated that because of lack of capital and of mutual coordination and cooperation among the smelters and the short-sighted policy of the different company directors there had been no marked progress in the metallurgy of antimony in China. The total smelting loss, now at least 20 per cent, due to the volatilization of the metal during the successive stages of treatment, should be reduced to 10 per cent or less. As a result of his experience, he recommended a number of important changes in smelting practice.

Market.—The market for antimony ores is confined to a few smelters in the United Kingdom and on the Continent and to the chemical industry in both Europe and the United States. The price is usually entirely a matter of private contract. In England, the smelting and refining of antimony ores has been long practiced by such companies as Cookson and Co., Ltd., Hallett and Son, St. Helens Smelting Co., Ltd., and others drawing their supplies of ore principally from China, Mexico and Bolivia. The French smelters operate principally on French and Algerian ores. The price paid for ores depends on the percentage content of antimony and of those elements considered as impurities because of their effect on the quality of the metal produced. Usually, sulfide ores are preferred, but oxide ores are also taken by some of the buyers, generally at a slightly lower price. The objectionable impurities are lead, copper, arsenic, zinc and bismuth. There is no definite ratio between the selling price of refined metal and the unit price of antimony ore. In all of the details relating to the base price for the metal and to the penalities for impurities, negotiation between buyer and seller based upon market conditions at the time, supply of and demand for ore, ultimately fixes prices and terms. In normal times, ample supplies of ore, practically free from the impurities named, are available, and consequently only such ores are salable. Weighing and sampling are done at the buyers' works and usually a 10-per-cent sample only is taken. Assays are compared and agreed and payment is usually made fourteen days from date of sampling, unless otherwise arranged.

The antimony market is one of the most unstable of the metal markets. The tonnage requirements are small and any extra quantity thrown on the market causes prices to react and the market practically to disappear. In New York in 1916 ordinary brands sold up to 45 cents per lb., while in 1922, at the bottom of the depression period, the New York price touched 4⅜ cents. In 1921, the price averaged 4.92 cents. The five year pre-war average was 7.43 cents. During the past year, 1924, owing to unsettled conditions in China and consequent light supplies in the United States and Europe, the New York price rose to 17½ cents at the close of the year and continued to advance early in 1925 to 22 cents, but, with more plentiful supplies in sight, it has now fallen to about 14 cents (March, 1925).

The imports of antimony into the United States during the year 1923 in tons of 2000 lb. (United States Geological Survey) were: ore (antimony content) 1047, crude 835, regulus 7813, oxides and other compounds 2277. Of the 7813 tons of regulus, 6593 arrived from China, 705 from England and 375 from Germany. The direct shipments from China were thus 84.4 per cent of the total arrivals and a large proportion of the imports from England and Germany were also of Chinese origin. In the five pre-war years 1909–1913, the average yearly imports into the United States of metal were 5241 tons, of crude and ore

1203 tons. During this same period the United States produced in the form of antimonial-lead a yearly average of 2260 tons contained antimony. It is thus evident that while there is no domestic mining industry in the United States there is a very considerable production of antimony from antimonial-lead ores. To this must be added the recovery in the form of secondary metal which amounted, in 1923, to 8021 tons, estimated equal to about 73 per cent of the total antimony imported. In the period immediately succeeding the war, large quantities of shrapnel bullets, etc., were also imported into the United States (entered under the tariff classification as type-metal), about 63,000 tons being imported from 1919 to 1922, inclusive, containing about 12,700 tons of antimony. The world's production in 1923 as reported by the United States Geological Survey in metric tons, was as follows:

WORLD'S PRODUCTION, 1923

	METRIC TONS
Mexico	490
United States	8
Bolivia	312
Austria	62
France	437
Italy	271
Spain	25
Jugoslavia	131
China	14,500
Turkey (Asia Minor)	400
Algeria	500
Victoria	421
Total	17,557

Uses.—Antimony is rarely used except when alloyed with other metals. A small quantity of the pure metal is used as a pigment, known as "Iron Black," for the purpose of bronzing metals and plaster casts and to produce the appearance of polished steel on articles of papier maché or pottery. The principal alloys of which antimony is a component part are type-metal, stereotype, linotype, Britannia metal, bearing metals, pewter, coffin trimmings, battery plates, hard lead, etc. Type metals usually vary in composition between the following ranges:

	LINOTYPE	MONOTYPE AND "SMALL TYPE"
Lead	83 – 85 per cent	64 – 78 per cent
Tin	4 – 5 "	6 – 12 "
Antimony	11 – 12 "	16 – 24 "

with sometimes a small percentage of copper in "small type." Britannia metals contain from 84 to 94 per cent tin, 15 to 5 per cent antimony, with small percentages of copper, zinc and lead. Bearing metals have a wide range of composition, usually being mixtures of lead, tin, antimony and copper; the antimony content varying from 5 to 18 per cent. Battery plates contain from

4 to 12 per cent of antimony while metal for coffin trimmings and other small castings contain 12 to 14 per cent of antimony, the remainder of these alloys being lead. For a complete list of alloys containing antimony, reference should be made to "A List of Alloys" prepared by William Campbell for Committee B-2 on Non-Ferrous Metals and Alloys.[1]

Other uses for antimony are in the form of its oxide, sulfide, chloride and other salts, in pigments, safety matches, fireworks, vulcanizing and coloring red rubber, preservation of wood and textiles and medicines.

The percentage consumption of antimony as given in the census of war commodities for 1917 and 1918, U. S. Department of Commerce is as follows:

	1917	1918
Babbitt Metal	29.7 per cent	28.1 per cent
Hard Lead, including pipe, traps, etc.	11.1 "	12.2 "
Soft-metal alloys and solder	8.9 "	10.2 "
Type and type metal, linotype, electrotype, stereotype	8.7 "	6.8 "
Vulcanized rubber and rubber goods	7.1 "	8.4 "
Shrapnel and other bullets	7.0 "	11.7 "
Bearing or anti-friction metals not elsewhere specified	6.6 "	6.5 "
Battery plates	6.1 "	4.8 "
Enamel on metalware	4.6 "	3.3 "
Cable coverings	2.7 "	1.1 "
Chemicals, paints and pigments	2.5 "	2.3 "
Brass, including bronze	1.0 "	0.8 "

Impurities.—The presence of impurities in the metal dulls the luster and tends to prevent the formation of large-size crystals. It is principally for this reason that the starlike appearance of the surface of the ingots is taken as an indication of purity.

The impurities, besides lowering the antimony content may cause loss in melting by formation of dross, or by volatilization. In the use of the metal for making alloys, any oxide or sulfide would act in this way. In melting at high temperatures, part of the arsenic might be lost by volatilization and part go into the alloy and the effect of the arsenic in the product would generally be detrimental. The presence of comparatively small amounts of arsenic, particularly in type and bearing metals, tends to make them brittle or friable, although it increases fluidity. Other impurities are usually not present in sufficient quantities appreciably to affect the alloys in which the antimony is used, unless their complete absence is required.

Analyses.—Each shipment of antimony received should be analyzed by the consumer, in order to be certain of its composition. Analyses showing amounts of impurities in some brands of antimony and methods of analysis are given in an article by W. A. Cowan,[2] from which the following is quoted:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 213 (1922).

[2] *Transactions*, Am. Inst. Metals, Vol. VIII, p. 196 (1914).

METALLIC ANTIMONY ANALYSES

All values are expressed in per cent.

	Cookson's "C"		Hallett's "H"		Japanese "MC"		Chinese "OO"	
	Laboratory A	Laboratory D	Laboratory A	Laboratory D	Laboratory A	Laboratory D	Laboratory A	Laboratory D
Lead	0.041	0.102	0.669	0.718	0.443	0.424	0.018	0.029
Tin	0.035	trace	0.175	0.012	0.175	0.012	0.035	none
Arsenic	trace	0.092	trace	0.210	0.008	0.095	0.017	0.090
Bismuth	.....	none	.....	none	.....	none	.....	none
Copper	0.040	0.046	0.038	0.046	0.034	0.043	0.008	0.012
Cadmium	.....	none	.....	none	.....	none	.....	none
Iron	0.010	0.004	0.014	0.007	0.015	0.007	0.007	0.004
Zinc	trace	0.034	trace	0.023	trace	0.023	trace	0.027
Ni and Co	.....	0.028	.....	none	.....	none	.....	trace
Sulfur	.....	0.086	.....	0.128	.....	0.201	.....	0.078
Antimony[1]	.....	99.608	.....	98.856	.....	99.195	.....	99.760
Total	.....	100.000	.....	100.000	.....	100.000	.....	100.000

Laboratory A: American Sheet and Tin Plate Co.
Laboratory D: National Lead Co.

[1] By difference.

As stated therein, the analyses represent only single ingots from lots purchased on the market at that time.

In the preparation of this article reference has freely been made to the following:

C. Y. Wang, "Antimony," Chas. Griffin and Co., Ltd., London, 1909; "Practice of Antimony Smelting in China," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., September, 1918, Vol. LX (1919); "The Mineral Resources of China," Tientsin, 1922.

Conference with the U. S. Tariff Commission on the Antimony Industry of the United States, San Francisco, June 24, 1918.

"Visit to Well-known Lead and Antimony Works," *Metal Industry*, London, May 4, 1923.

United States Geological Survey, "Antimony in 1923," by Frank C. Schrader. "Secondary Metals in 1923," by J. P. Dunlop.

W. A. Cowan, "Metallurgy of Tin and Antimony," *Transactions*, Am. Inst. Metals, Vol. VIII (1914).

"Metal Statistics," *The American Metal Market*, New York City.

Bulletin of the Imperial Institute, Vol. XIV, No. 3, July, September, 1916.

"Antimony" (1913–1919), Imperial Mineral Resources Bureau (1921).

Lawrence Addicks, "A Review of the Tin Situation," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 184 (1923).

William Campbell, "A List of Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 213 (1922).

H. K. Masters, Various articles on Antimony in *Mineral Industry* and *Engineering and Mining Journal Press.*

ADDENDUM TO REPORT OF COMMITTEE B-2

At a meeting of Committee B-2 held in Atlantic City on June 23, 1925, action was taken to rectify an error in the Tentative Specifications for Manganese-Bronze Ingots for Sand Castings (B 7 - 24 T). It was originally intended that a footnote appear in these specifications referring to the use of virgin metal in manganese, and the inclusion of such a footnote had been approved by Committee B-2 in 1924. The report of the committee for that year failed to provide for the inclusion of the footnote in the tentative specifications. Committee B-2 accordingly took action at the meeting to include a footnote to the central heading "Chemical Properties" in the specifications to read as follows:

> "Because of claims made of the improved qualities of manganese bronze made from virgin metal (copper conforming to the Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (Serial Designation: B 5) and zinc conforming to the requirements of Grade 2 or better of the Standard Specifications for Spelter (Serial Designation: B 6) of the American Society for Testing Materials, 1924 Book of A.S.T.M. Standards) the buyer may, at his option, elect when contracting that these materials only be used."

Respectfully submitted,

William Campbell,
Chairman.

P. D. Merica,
Secretary.

Editorial Note

The proposed revisions of the Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars; for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars; for High Sheet Brass; for Seamless Admiralty Condenser Tubes and Ferrule Stock; and for Naval Brass Rod for Structural Purposes, referred to in the report, were accepted for publication as tentative and appear on pages 898-903.

The Tentative Specifications for Seamless 70-30 Brass Condenser Tubes and Ferrule Stock; and for Seamless Muntz Metal Condenser Tubes and Ferrule Stock were approved at the annual meeting and subsequently adopted as standard by letter ballot of the Society on August 28, 1925, and appear in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

The proposed revisions of the Tentative Specifications for Light Aluminum Casting Alloys; and for Aluminum Ingots for Remelting for Rolling; and of the Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys were accepted. The tentative standards as thus revised appear on pages 574, 585 and 590, respectively. The proposed Tentative Specifications for Muntz Metal Condenser Tube Plates were accepted for publication as tentative and appear on page 583. The correction in the Tentative Specifications for Manganese-Bronze Ingots for Sand Casting, mentioned in the Addendum to the report has been made in the specifications as printed in the 1925 Book of A.S.T.M. Tentative Standards.

REPORT OF COMMITTEE B-3
ON
CORROSION OF NON-FERROUS METALS AND ALLOYS

During 1924, Committee B-3 has been active in starting actual laboratory tests and in making necessary revisions in the methods of testing as outlined in last year's report.

Materials for Tests

The work of cutting up the large sheets of material into test specimens proved to be too much to expect of the Bureau of Standards and the sheets were returned to the companies who supplied them. In every case these companies cooperated whole-heartedly and specimens cut to the required size were sent back to the Bureau of Standards for distribution.

The Bureau of Standards summarized the information received from the manufacturers with regard to their materials. A specimen of each material was analyzed at the Bureau of Standards. Tensile properties of each of the materials were determined upon specimens cut longitudinally and transversely. The microstructural characteristics of the materials were studied and micrographs were made which are believed to be representative of the various materials.

After distribution of specimens to the cooperating laboratories the following specimens are still available at the Bureau of Standards:

1770	Lead	1415	Admiralty Metal
1390	Nickel	1480	Copper
1780	Zinc	244	Aluminum

The last two laboratories cooperating in the tests were not supplied with aluminum samples because of the shortage of this material. The shortage was due to scratches, etc., discovered on the aluminum sheets when they were returned to the Aluminum Co. of America for cutting up into specimens. Those portions of the sheets which were badly scratched had to be discarded and only the balance used for specimens.

Preparation of Specimens for Tests

It was decided that specimens for test shall be prepared within twenty-four hours of the starting of the test and preserved in a desiccator until actual start of the test.

In the case of aluminum specimens the emery papers may be prepared by the adding of a solution of paraffin in kerosine.

Letters have been addressed to each of the suppliers of the metals used by the committee, requesting them to study methods of cleaning their metals while making their tests and to report the results of their studies as soon as possible.

SUB-COMMITTEE I ON TOTAL IMMERSION

Sub-Committee I, W. S. Calcott, chairman, presented the following questions to the committee:

1. Whether or not solutions should be changed after the first 48 hours' immersion of test specimens.

   It is recommended that this be done, so that the corrosion rate should be determined by the action of fresh solution on test specimens prepared by previously immersing in a solution of the same initial composition.

2. A number of the test specimens were received with deep scratches and stamped marks upon them.

   It is recommended that all those of this type be discarded and fresh samples secured from the Bureau of Standards, and that the minor scratches be ground out even where the extent of grinding is sufficient to cause a distinct alteration in the size of the specimen.

3. In connection with the preceding, it is recommended that each sample be measured before testing so that the accuracy of the results will not depend upon the adherence of the specimen to the prescribed dimensions.

4. Determination of pitting corrosion:

   It is recommended that, in view of the uncertainty of the relative accuracy of the various methods proposed, each laboratory determine pitting corrosion by as many of the three suggested methods as feasible and report all three. The three methods are by the use of a ball point dial, by microscopic measurement, and by grinding out the pits and reweighing. The final choice of a method should be based upon the results of these measurements.

5. Determination of edge pitting:

   While it is recognized that the edges of practically all test specimens will pit more than the faces, it is recommended that edge pitting be ignored in estimating corrosion, on the ground that in the actual construction of chemical apparatus the edges of the constituent plates are not exposed.

6. On the question of the use of Pyrex apparatus for the handling of solutions for the total immersion test, for the sake of uniformity it is recommended that this glass be adhered to.

SUB-COMMITTE II ON ALTERNATE IMMERSION TESTS

Sub-Committee II on Alternate Immersion Tests, R. J. McKay, chairman, discussed the following subjects:

Method of Cleaning Samples.—In the alternate immersion tests, some difficulty will be found in removing the adhering corrosion product from the sample, so as to obtain reliable weight difference and measurement. It is our suggestion that the manufacturer of each metal be asked to suggest a method for chemical cleaning of the metal. This method should be submitted to Committee B-3 and acted on by it. The making of tests should not be delayed waiting for these results, but should go forward in the meantime, each laboratory using its best judgment in cleaning.

Total Testing Time.—The time suggested in the original specifications should be changed as follows:

Tests in dichromate and salt solutions should be made first for a period of 3 days. If definite change in weight is not obtainable in that time, the test should be repeated until definite change in weight is obtained.

In other solutions, tests should be made for 24 hours, and repeated until definite change in weight is obtained.

Renewal of Solutions.—Solutions should be changed every day during the tests.

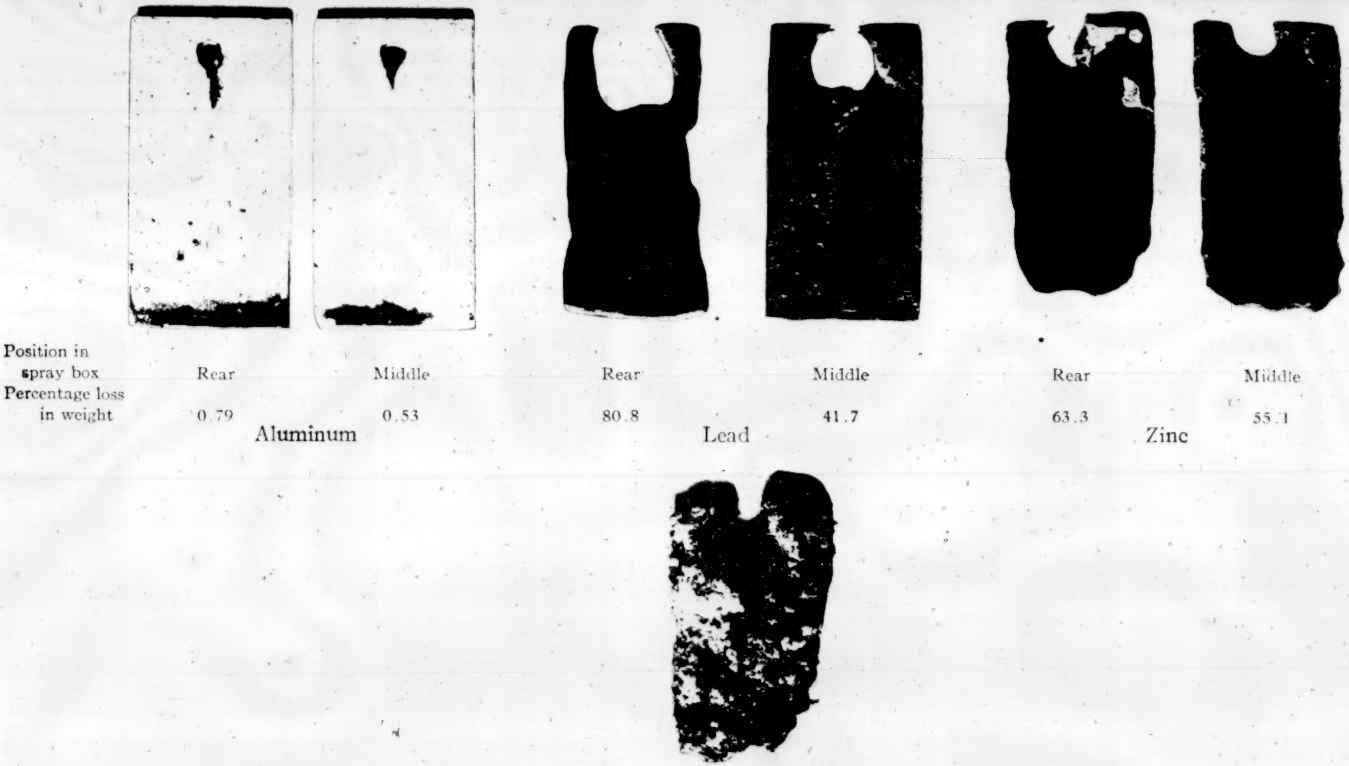
Area of Samples.—The area of samples as furnished is not always within specifications and considerable grinding will have to be done to put them in shape, which will further change the area. It is our suggestion that each sample be measured and its area calculated.

Reports of Tests.—Results as obtained should be reported on the test sheets to the chairman of the sub-committee, who will tabulate them and report to the main committee.

Retention of Samples Pending Decision on Results.—After tests are finished, the samples should be held by the laboratory making the test, in as nearly as possible the same condition, until decision is made as to whether the results are complete or further tests will have to be made.

SUB-COMMITTEE III ON SPRAY TEST

Sub-Committee III, H. S. Rawdon, chairman, has drawn up more definite instructions for carrying out of the spray test to supplement those given in the 1924 report of the committee. The experience


Position in spray box
Percentage loss in weight
Rear
0.79
Middle
0.53
Aluminum
Rear
80.8
Middle
41.7
Lead
Rear
63.3
Middle
55.1
Zinc

Zinc, with deposit left intact,
13 days exposure.

FIG. 1.—Specimens Exposed to Spray Test with Normal Acetic Acid. Showing Variation in Rate of Corrosion with Position in Box.

The black patches on the aluminum specimen are due to deposits of copper from the sprayed solution which had become contaminated with corrosion products resulting from the attack of specimens of other types of material exposed at the same time in the spray box.

of several members with the salt-spray test was used as a basis. These recommendations, in brief, provided for putting all the six different kinds of metal in the box at the same time, fresh solution in 2-liter lots to be added every day.

The experience of the Bureau of Standards with the acetic acid spray test, carried out in this manner, showed clearly the urgent need for a further modification and revision of the method for conducting this test. Fig. 1 illustrates the two main difficulties encountered.

The first difficulty is the electrodeposition of the more "noble" metals, in this case copper, upon the less "noble," for example, aluminum. The solution within the spray-box soon became contaminated with copper by the rather rapid attack of the copper by the acetic acid. The mist sprayed from this contaminated solution contained copper and deposition of metallic copper was readily detected on the zinc, lead, and aluminum. This observation shows the need for preventing the contamination by the products of corrosion of the stock solution to be sprayed. This difficulty was not experienced with the sodium chloride spray, however.

The second difficulty encountered is the variation in the rate of corrosive attack according to position of the specimens within the box.

Another observation on the very rapid attack of certain metals makes the shortening of the period between weighing (two weeks was originally decided upon) very desirable.

The following procedure for carrying out the spray test was drawn up to supplement that given in the 1924 report of the committee and to supersede any previous recommended practice in any cases where conflicts occur.

1. The spray box must be modified so as to prevent the contamination of the solution to be sprayed by the corrosion product draining back from the exposed specimens. (This may be done fairly easily in either one of two ways. The tube of the "atomizer" through which the solution is drawn up to be sprayed may be lengthened and passed through a hole in the floor of the box into the stock solution which would thus be kept entirely outside of the box. A second possible method necessitates installing a "dam" across the sloping floor of the box to catch the corrosion product as it drains back and thus prevent contamination of the solution to be sprayed, the solution being contained in the lower part of the box as heretofore and the atomizer being unchanged. This is the plan which is being tried out at the Bureau of Standards, since by extending the "dam" up as a high baffle plate, it can be made to help serve two purposes (see recommendation 2 below).)

2. Care must be taken to insure uniform corrosive attack of all the specimens of a kind which are in the box at the same time. Swirling currents in the mist can be minimized by the use of a transverse baffle plate near the "atomizer" to within a couple of inches of the top of the box. The position of specimens in the box should be changed frequently; if the run is more than 24 hours, they should be changed every day.

3. All the specimens are to be put into the box at the same time as hitherto. Zinc, lead, and aluminum specimens in acetic acid, hydrochloric, sodium hydroxide, and ammonium hydroxide are to be removed, washed (except as mentioned in recommendation 4), dried, and weighed at the end of 24 hours. This will be repeated until a loss of 100 mg. is noted. For the other metals in these solutions and for all metals in the other solutions (sodium chloride and potassium dichromate) 72-hour periods are to be used. If for any metal and solution the loss in 24 hours is *very* slight, the 72-hour period may be substituted, record being made of the change. The test in any case need not be continued beyond 30 days, that is, a total immersion of 30 days, unless desired by the operator, a record being made of the results at the end of the 30-day period.

4. For aluminum and zinc, the two series of specimens, heretofore recommended should be continued, one to be weighed with and the other without removing the corrosion product, for the sodium chloride and potassium dichromate solutions. The same procedure for other metals and solutions may be carried out as the operator may desire, since sufficient specimens will be available. Cleaning should be done mechanically, a stiff bristle brush being permitted for this.

5. The measurement of depth of pits will be made at the conclusion of the test, that is, when 100 mg. loss has occurred or when the test has been discontinued (30 days).

6. The solution should be renewed daily as heretofore, 2 liters being used. It will not be necessary to follow the procedure outlined in the 1924 report of Committee B-3 for the preliminary purification of the chemicals. C. P. materials may be used directly.

7. The air purification train placed between the source of air supply and the spray box may be simplified by the removal of the wash bottles, if desired. The soda lime tower and the cotton wool oil filter are to be retained.

8. The corrosion product should be removed *frequently*, preferably daily.

SUB-COMMITTEE V ON ACCELERATED ELECTROLYTIC TEST

Sub-Committee V on Accelerated Electrolytic Test, J. S. Fuller, chairman, has received some very interesting results presented by Mr. Hall of the Bureau of Mines on corrosion tests made in mine water, both with and without impressed current. From these experiments, Mr. Hall concluded that the corroding agent in mine water is ferrous sulfate.

It has been brought out by the members of Sub-Committee V, who have commenced work on the accelerated electrolytic test, that the method of joining the No. 20 copper supporting wires to the samples, namely, soldering, was not satisfactory, especially with the aluminum and lead samples. It was, therefore, recommended that the supporting wires be mechanically joined to the samples by forcing them into a fine saw slot or hole drilled in the samples instead of by soldering.

The members of Sub-Committee V, who are at work, reported certain difficulties and expressed doubt about the magnitude of current, and the length of time of immersion, as outlined in the present accelerated electrolytic test. Doubt was also expressed concerning the suitability of this test for metals such as aluminum which readily form insoluble, non-reducible films.

Because of the very general lack of agreement, it has been decided that the laboratories which have already started this test should continue as previously outlined, but that those which have not should follow the recommendations of a committee on procedure consisting of the following: F. L. Antisell, *Chairman*, M. F. Sayre, and H. S. Rawdon. This committee will outline a modified Accelerated Electrolytic Test, and report to Sub-Committee V as soon as possible.

PITS

The following definition of the word "pit" was adopted: "A pit is defined as an abrupt depression."

The committee has considered various methods of determining the depth of pitting as follows:

1. Grinding down of the face of the specimen to the bottom of the deepest pit entails too much work.

2. Measuring the depth of pits mechanically by means of a depth measuring micrometer ground down to a sharp point or point of small radius is subject to too great an error and possible misleading data due to the possible penetration of the point of the micrometer into the softer metals.

3. A focusing micrometer or depth measuring adjustment on the ordinary laboratory microscope is the easiest of application to the problem in question.

Committee B-3 has decided that any method of grinding is satisfactory for the rough grinding operations and that only the finishing need be done in accordance with the instructions previously laid out except that "emery paper shall be backed up by felt or other soft cushion material so as to perfect a uniform grinding action on the face of the specimen being prepared."

The committee recommends that pits be measured to the nearest 0.005 mm. and that data sheets show the approximate number of pits per square centimeter of surface, the depth of the deepest pit, and the average depth of pitting.

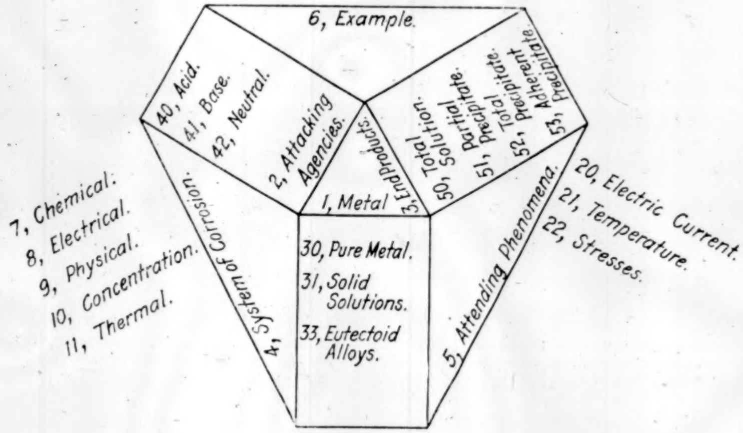

6, Example.
40, Acid.
41, Base.
42, Neutral.
2, Attacking Agencies.
3, End Products.
1, Metal
50, Total Solution.
51, Partial Precipitate.
52, Total Precipitate.
53, Adherent Precipitate.
7, Chemical.
8, Electrical.
9, Physical.
10, Concentration.
11, Thermal.
4, System of Corrosion.
30, Pure Metal.
31, Solid Solutions.
33, Eutectoid Alloys.
5, Attending Phenomena.
20, Electric Current.
21, Temperature.
22, Stresses.

FIG. 2.

TEST RESULTS, RATE OF CORROSION

The committee has decided that all test results are to be computed both in terms of centimeters penetration per month and in milligrams change in weight per square centimeter per twenty-four hours.

The committee has also decided to recommend to the Bureau of Standards the methods of reporting and measuring corrosion as they now appear on the Laboratory Record Forms, this being done so that the Bureau of Standards may officially consider them in connection with its work with the National Research Council.

DEFINITIONS

To comprehensively define the attendant phenomena of corrosion, it is self-evident that the common usage of the term corrosion should demand attention. If defined in the following manner the term would become so broad as to assume added importance.

Corrosion.—The phenomena of corrosion, due to, accompanied by, or resulting in, a chemical change.

The three fundamentals of corrosion are:

1. The metal;
2. The attacking agency;
3. The end products.

These three considerations are associated with:

1. The system of corrosion;
2. The attendant phenomena of corrosion.

The tentative diagram shown in Fig. 2 is suggested with the bare possibility of determining the relationship of attending factors, provision being made for showing examples of corrosion. The subdivision of each of these captions would bring into relationship a great number of combinations, for many of these combinations present no ordinary terms.

Various systems of corrosion could be noted on separate sheets in the hope that we would be fortunate enough to correlate the phenomena of corrosion in different systems.

The terms of secondary importance as used in the manipulation of corrosion investigation will follow the terms already used by Committee B-3, and be compiled in the regular manner. For the standardizing of terms to be hereafter coined, Sub-Committee IV on Definitions, F. L. Anitisell, chairman, will make tentative recommendations that it believes should be reported to the committee as a whole for study and consideration.

This report has been submitted to letter ballot of the committee, which consists of 40 members, of whom 35 have voted affirmatively, none negatively, and 5 have refrained from voting.

Respectfully submitted on behalf of the committee,

E. C. LATHROP,
Chairman.

SAM TOUR,
Secretary.

REPORT OF COMMITTEE C-1
ON
CEMENT

Since the last annual meeting of the Society, Committee C-1 on Cement has held two meetings. It has undertaken the detailed review of the standard specifications and methods of tests for cement to determine what changes, if any, are desirable in the light of current practice or of the results of investigations by its sub-committees and others.

The committee believes its functions to be not only investigative but judicial; that is, its duty is to conduct investigations and to review current practice and to express improvements in this practice through its recommended specifications and methods of tests for cement.

The committee calls attention to the fact that the present Standard Specifications and Tests for Portland Cement (C 9 – 21)[1] are the result of intensive studies of current practice and of the results of the investigations by its sub-committees. Each requirement of the present specifications is based upon substantial data which has been published by the Society. Since the adoption of the Standard Specifications for Portland Cement, Committee C-1 through its sub-committees has continued its study and investigation of specifications and methods of test for cement. The committee believes that revisions of these specifications should also be based upon conclusive data. Committee C-1 records its firm belief that the present Standard Specifications for Portland Cement provide a material which, if properly used in accordance with recognized good practice in making concrete, will produce a concrete of high quality.

It is the opinion of the committee, based on investigations of results obtained at a large number of laboratories, especially interested in testing cement, that the great need at the present time is better concordance in results by different operators when using standard specifications in testing cement. To aid in this it has prepared a Laboratory Manual which will point out the cause of some variations and through an interpretation of methods of tests contained in the standard specifications will it is hoped result in a more uniform practice among laboratory operators. Committee C-1 submits this manual, appended hereto, as information and with the request that it be printed for distribution with the Standard Specifications for Cement.

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[1] 1924 Book of A.S.T.M. Standards.

Committee C-1 has approved the recommendation of its Sub-Committee on Specific Gravity that the requirement for specific gravity, now an optional requirement in the Standard Specifications for Portland Cement (C 9 – 21) be eliminated and submits this revision as information.

Committee C-1 also submits as information revisions of the Standard Specifications for Natural Cement (C 10 – 09).[1] The proposed revisions of the present specifications are:

1. The elimination of the neat cement requirements;
2. Changing the requirements for fineness and time of setting so as to conform to the requirements for fineness and time of setting of the Standard Specifications for Portland Cement; and
3. The elimination of any specific reference to the weight of the package but providing that the weight be plainly stamped on each package.

This report has been submitted to letter ballot of the committee, which consists of 59 members, of whom 42 have voted affirmatively, 1 negatively, and 16 have refrained from voting.

Respectfully submitted on behalf of the committee,

R. S. GREENMAN,
Chairman.

R. B. YOUNG,
Secretary.

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[1] 1924 Book of A.S.T.M. Standards.

APPENDIX

MANUAL OF CEMENT TESTING

Supplementing the Standard Specifications and Tests for Portland Cement (C 9 - 21) of the American Society for Testing Materials

INTRODUCTION

Portland cement is an important structural material which is manufactured and sold in large quantities under a uniform specification. The tests of cement are essential features of the specification; faulty test methods are certain to lead to erroneous conclusions as to the quality of the cement. Many steps are involved in making cement tests and the results are greatly influenced by seemingly minor variations in procedure.

Cement testing is not an exact science. Existing test methods are, at best, crude tools and it must not be expected that exact agreement will be secured in tests by different operators or by the same operator at different times, even when made on the same sample and under the same conditions.

It is the purpose of this manual to emphasize those factors which may affect results of tests and to call attention to less apparent influences which are important, but are sometimes overlooked.

The recommendations of this Manual refer to the Standard Specifications and Tests for Portland Cement of the American Society for Testing Materials (Serial Designation: C 9 - 21).[1] They do not in any way supercede the standard tests, but are intended, rather, to supplement the physical tests and to suggest a procedure in certain sections where considerable latitude is permitted by the standards and to recommend methods which the committee has found to be satisfactory and conducive to greater uniformity.

The specifications for portland cement are intended to set forth minimum requirements which must be met. Due to the wide range in conditions under which tests are made and the varying experience of operators, it is obvious that a certain latitude must be allowed for differences in test results. It may be expected that if the test procedure is carefully followed and if the testing equipment is in good condition experienced operators will secure tensile strengths well above minimum requirements of the specifications; however, if careless methods are used and numerous short-cuts are made, strengths may be found to fall below requirements and lead to rejection of a good product. If 20 different operators make tests on the same cement and 15 of them find strengths well above specifications and 5 find strengths below, it is obvious that the latter have not properly standardized their tests.

Cement testers should be familiar with the Standard Specifications and Tests for Portland Cement of the A.S.T.M., and should use every care to follow standard methods strictly.

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[1] 1924 Book of A.S.T.M. Standards.

A mass of information on many of the properties of portland cement, including variations in tests, is given in "Data Considered by Committee C-1 of the American Society for Testing Materials in preparing the Standard Specifications and Tests for Portland Cement," published by the committee in 1919.

It is suggested that different cement testers in a given city or district arrange to carry out tests on the same cement and exchange results. This procedure will do more than anything else to create an interest in standard methods and will be found effective in eliminating erroneous practices and in calling attention to defective apparatus. A suggested course of training for cement testers is given on pages 170 and 171.

To encourage a more intimate knowledge of the literature on portland cement, a selected bibliography is appended, listing some of the better known and more important sources of information on the subject. The bibliography is general in scope, but those who are primarily concerned with the testing of cement will find references to this phase of the subject grouped under the sub-divisions, "Physical Properties and Tests" and "Specifications."

SAMPLING

(See Sections 11, 16, 17, 18, 19 of the Standard Specifications and Tests for Portland Cement (C 9 – 21) of the American Society for Testing Materials.[1])

1. *Size of Sample.*—The specifications require that test samples shall weigh at least 8 lb. The test sample may be an individual sample or a composite sample. If tests are to be made on individual samples, each sample must weigh at least 8 lb.; if tests are to be made on composite samples, the individual samples must be of such size that when composited, the composite sample will weigh at least 8 lb.

2. *Number of Samples (Car samples).*—The specifications permit the testing of either individual or composite samples from cars. In the former case a test sample is taken for each 50 barrels or fraction thereof. In the latter method a sample must be taken from one sack in each 40, and all samples so taken composited to form a test sample.

3. *Number of Samples (Bin samples).*—Either individual or composite test samples may be used in testing a bin. Individual test samples must be taken from each 100 barrels; composite test samples must consist of at least 2 individual samples, and must represent not more than 200 barrels.

4. *Treatment of Samples.*—The sample should be marked plainly and identified completely on the outside of the container as to source and date of sampling. Also, a complete identification should be enclosed in the container. Samples should be forwarded to the laboratory promptly, preferably in air-tight containers. Cement samples absorb moisture and carbon dioxide much more rapidly than packages of standard size, because of the relatively larger surface exposed. Furthermore, the rate of absorption varies under different conditions. It is important to keep these variable influences at a minimum.

5. Prior to testing the samples must be passed through the No. 20 sieve, and all foreign matter and lumps which do not pulverize easily in the fingers should be discarded.

Reference.—J. R. Dwyer and Roy N. Young, "Inspection and Testing of Portland Cement," *Concrete*, Vol. 21, pp. 56 and 95, August and September, 1922.

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[1] 1924 Book of A.S.T.M. Standards.

DETERMINATION OF FINENESS

(See Sections 32, 33, 34, 35, Specifications C 9 – 21.)

6. *Accuracy of Tests.*—Accuracy in sieving is required in all standardization and check tests, in comparative and routine tests. In order to make fineness determinations satisfactorily, the operator must possess considerable skill, which can be acquired only by practice. With all the care that can be taken, he is a good operator who can check himself regularly within a range of 0.5 per cent on portions of the same thoroughly-mixed sample and on the same sieve in triplicate tests. In general, it may be stated that if an accuracy of 1 per cent or better is required, it is necessary that standard calibrated sieves be used. Because of unavoidable variations in the wire cloth, a small correction is generally necessary in order to bring the performance of commercial sieves into line with a standard sieve.

7. *Certified Sieves.*—Certain sieve manufacturers supply "certified sieves" with correction factor determined by the U. S. Bureau of Standards, Washington, D. C. This correction must be *added* to the *percentage passing* the sieve when it has the plus sign; it must be *subtracted* from the *percentage passing* the sieve when it has the minus sign. Sieves having a correction factor of more than ±3 per cent should not be used. An occasional redetermination of the correction is desirable as an indication of the satisfactory condition of the sieve.

8. *Standard Fineness Samples.*—For the benefit of those who wish to determine the correction factors of their sieves the Bureau of Standards furnishes samples of cement of standard fineness at 50 cents each. These samples are mailed in sealed glass bottles containing about 160 g. of cement (enough for 3 tests) and are guaranteed to be within 0.2 per cent of the true fineness as determined on the Bureau's standard sieves. A more general use of standard fineness samples is recommended, not only for determining corrections to sieves, but also for checking the performance of operators, and for preparing and standardizing auxiliary fineness samples for use in the laboratory.

9. *Machine Sieving.*—Machine sieving usually gives different results from hand sieving. For accurate machine sieving, therefore, it is necessary to determine a correction for sieve and machine together and to apply this correction, rather than the correction of the sieve alone, to the result obtained.

10. *Design of Sieves.*—It is essential that all sieves used in the determination of fineness of cement be well designed and constructed and that they be provided with accurately fitting covers, in order to avoid the loss of small quantities of material during the sieving operation.

11. *Balance.*—A good balance of sufficient sensitiveness should be used. To determine the end point of the operation where 0.05 g. passes through in one minute of continuous sieving, the balance should be reliable to one or two milligrams for best results.

12. *Method of Testing.*—The rate and uniformity of the sieving motion is very important, especially during the one-minute final check tests. The sieve should be operated with a gentle motion, uniformly, and as close to the specified 150 times-per-minute as possible, keeping the sample well spread out on the sieve. A timepiece of some sort is indispensable. A watch, with a second hand, hanging on the wall in front of the operator is convenient and satisfactory. The sieve should be rotated throughout the process as specified, particularly in the one-minute test. The rotation is essential to average the irregularities in the wire cloth.

13. *Loss of Material.*—Loss of material must be guarded against. It is not unusual for beginners to lose residue without suspecting it until the habit of uniform, gentle motion and tapping has been acquired. Sieving over a white paper will detect such loss. Residue is not infrequently caught in the seams or crimping of poorly designed or poorly constructed sieves and covers during either the sieving or the cleaning process. Poorly fitting covers may permit loss of material during sieving. Sieves and covers should be examined to see that loss may not occur in this way. Sieves should also be frequently examined for defects in the cloth.

14. *Cleaning Sieves.*—Sieves should be kept thoroughly clean and dry. Dust and residue particles should be brushed from the sieve by means of a brush with bristles stiff enough to clean the sieve but not heavy enough to injure it. A 1-in. bristle brush with an 8 or 10-in. handle is very convenient for this purpose.

15. *Humidity.*—Excessive humidity and damp samples interfere with sieving and tend to decrease the percentage of cement passing the sieve. Important tests should be avoided on days when the relative humidity is high.

Reference.—"Standardization of the No. 200 Cement Sieve," U. S. Bureau of Standards *Technologic Paper No. 42.*

MIXING CEMENT PASTES AND MORTARS

(See Sections 36 and 37, Specifications C 9 – 21.)

16. *Mixing Table.*—The height of the mixing table from the floor has a surprisingly large influence on the efficiency of the mixing and molding operation. A height of about 36 in. is correct for the average operator. Unusually tall men require a little more than this, and unusually short men a little less. The table top should be plain and made of non-absorbent material. Plate glass, though not essential, makes the best all-around mixing table top. With ordinary care, plate glass ½ in. in thickness should prove entirely satisfactory. The mixing table must be kept clean—free from adhering cement, oil, etc.

17. *Balance.*—The use of a good balance is recommended. It should be sensitive to 1 g. and should be kept clean and in good order at all times. Single 250, 500, and 750-g. weights may be used both for convenience and to minimize errors in reading of weights on scale pan.

18. *Graduates.*—The mixing water should preferably be measured in a graduate having a capacity not more than twice the volume of water required in the mix. A 200 or 250-cc. cylindrical graduate, with graduations reading in both directions, is recommended. When a new graduate is used, it should be checked at several points to determine its accuracy. When measuring the water in a graduate, the reading is taken by holding the graduate vertical and sighting through it along the bottom of the meniscus, or the surface of the water at the center, and not at the outer edge of the meniscus where the water wets the glass.

19. *Gloves.*—Rubber gloves must always be used for mixing cement pastes and mortars. Gloves should be flexible enough so as to permit of easy movement of the fingers and should be provided with cuffs. Comfortable and satisfactory gloves are by no means unimportant matters in cement testing. After using, gloves should be well cleaned, and if they are of the cloth-lined variety they should be turned partly inside out when removing from the hands to permit drying of the interior. This will prevent rotting and unpleasant odor due to

perspiration. Oils and grease will injure the rubber. Gloves containing large holes should not be used.

20. *Size of Batch.*—A 500-g. batch should always be used for neat cement and a 1000-g. batch for mortar. The size of the batch is closely related to the possible thoroughness and uniformity of mixing, other conditions being the same, and is therefore not without its effects on the results of the tests. Small batches may lose relatively large amounts of water by evaporation, and by loss on gloves and slabs. On the other hand, with the same amount of time and mixing much larger batches may not be so thoroughly or so uniformly mixed. Longer mixing would probably be required for the larger batches, and another variable would be introduced. Moreover, the longer time required to mold the larger batches into specimens may result in considerable evaporation of water, stiffening of the cement through setting action and an accompanying decrease in the ease of molding and in the uniformity of the results.

21. *Dry Mixing.*—The batch must be thoroughly mixed dry, prior to the introduction of the water. The material should not be scattered widely, but neatly and thoroughly mixed by turning the materials with the trowel, and by rubbing them lightly upon the mixing table with the hand until the mass shows by its uniform appearance that the ingredients are well mingled. This method will cause less segregation than rolling the dry materials on a paper. Then a crater which will hold the entire desired quantity of mixing water without overflow or loss should be formed in the dry materials.

22. *Mixing Water.*—In general, water fit for drinking is satisfactory for cement testing. The amount of water present in a mix is of great importance, particularly with regard to the setting time and strength. Water largely in excess of the required quantity will prolong the setting time and lower the strength. Less than the proper amount of water may shorten the setting time and result in strength variations from the normal, the latter sometimes due to the difference in molding properties of the mortar. The amount of water used is expressed in cubic centimeters (1 cc. of water = 1 g.), and is usually measured by volume. The range in variation in the amount of water required by most portland cements for the 1:3 standard sand mortar mixes is not large, and the water must be carefully measured. The amount of water in a mix may be unintentionally increased by using wet gloves, or by pouring the dry materials on a wet slab. Loss of water may be somewhat avoided by commencing the mixing of the dry materials and the water so as to spread as little as possible of the mix over the surfaces of the gloves and slab.

23. *Addition of Mixing Water.*—The whole of the mixing water must be poured at one time into the crater in the dry materials. The dry materials should then be turned, as quickly as possible, from the outside into the crater, by means of a trowel, covering all the water. After the dry material has been thus turned into the water, one-half minute must be allowed for the absorption of the water before mixing is commenced. This one-half minute absorption interval must be well observed.

24. *Operation of Mixing.*—Thorough, continuous, and vigorous mixing immediately following the half minute allowed for absorption of the water is essential to produce well and uniformly mixed pastes and mortars. Increase in the rate of mixing, or the time, will often produce increase in workability, or may effect greater strength. If, however, mixing is carried to a point where evaporation of water and reaction within the cement lessens the moisture and

workability to an appreciable extent, the time of set specimens may be drier than normal, and the strength specimens cannot be so easily molded. A uniform method should be adopted in which an endeavor is made to put sufficient effort into the operation to accomplish the necessary amount of mixing in one minute. A timepiece should be used for these operations.

25. *Temperature and Humidity.*—The temperature of the room, the dry materials, the mixing table and the apparatus, should be maintained within ±5° C. (10° F.) of 21° C. (70° F.). The temperature of the mixing water should be maintained within ±3° C. (5° F.) of 21° C. (70° F.). Temperature and humidity have a great effect on the behavior of the mixes. Increase in temperature is accompanied by decrease in setting time, and usually by acceleration of strength. Drafts of air and relatively low humidity will cause rapid evaporation of water from the mixes. When the relative humidity of the air is low, say below 50 per cent, the moisture in the laboratory may be increased somewhat by sprinkling water on the floor.

NORMAL CONSISTENCY

(See Sections 38, 39, and 40, Specifications C 9 - 21.)

26. *Vicat Apparatus.*—The Vicat apparatus should be inspected frequently. The weight and diameter of the rod should be checked to make sure that it moves freely and is free from rust and cement. (See Paragraph 35.) Faulty results in determination of normal consistency are frequently due to defective or dirty apparatus.

27. *Method.*—Exactly 500 g. of cement must be used in the determination of normal consistency. For details of mixing, see Paragraphs 22–25, inclusive. If a trial paste is too dry it should not be remixed with more water, but should be discarded. A new sample should be taken in order to avoid errors due to evaporation and other causes.

28. *Care in Determination.*—The accuracy of the normal consistency determination depends upon the proper placing of the cement paste in the mold, removal of the surplus with as little disturbance of the mass as possible, freedom of the apparatus from vibration, and careful manipulation throughout the test. The operator must possess considerable skill and dexterity in order to make this test satisfactorily. It is a very important determination, however, because upon it depends the percentage of water which is used for all the other determinations.

DETERMINATION OF SOUNDNESS

(See Sections 41, 42, 43, 44, Specifications C 9 - 21.)

29. *Moist Closet.*—A well-made moist closet, or moist cabinet, is essential for use in cement testing in order to maintain a high degree of relative humidity. Pans of water in the top of the cabinet and pieces of wet wicking on the sides and such similar methods as will bring large areas of wet surface in contact with the air of the cabinet are desirable features. The use of a wet and dry bulb thermometer is recommended as a means of determining the relative humidity. The doors should be well fitted and should be kept closed at all times except during the actual operation of introducing or withdrawing specimens. It is desirable to have separate compartments for the time-of-set specimens and the briquettes, because of the necessity for frequently opening and closing doors in the case of the compartment for time-of-set specimens in order to make

determinations. The temperature of the moist closet should be maintained within ±3° C. (5° F.) of 21° C. (70° F.). This may be accomplished either by means of an automatic temperature control device or the temperature may be regulated by the use of warm water or ice.

30. *Steaming Apparatus.*—The steaming apparatus should be capable of maintaining a constant temperature of 98 to 100° C. for 5 hours. The introduction of large quantities of water during the test, thereby lowering the temperature, should be avoided. A simple overflow or other constant level device should be used.

31. *Size of Batch.*—Not less than 500 g. of cement should be mixed with the hands using the quantity of mixing water determined by the normal consistency test. Since the specified minimum mix of 500 g. makes considerably more paste than is required to make a pat, 100 and 200-g. samples are sometimes mixed with a trowel or with the hands. This method usually requires more mixing water than that specified by the normal consistency test, which changes the rate of hardening of the pat. Results obtained in this manner are not standard and should be considered as only approximate. The shape and size of the pats should be as specified. Small pats or pats with blunt edges do not readily show the effects of slight unsoundness.

32. *Time in Moist Closet.*—The pats should remain in the moist closet 24 hours. The degree of hardening attained by the pats at the moment of placing in the atmosphere of steam is important. Unsound pats may appear sound if steamed when less than 24 hours old; and pats which comply strictly with the specification may appear unsound if steamed when more than 24 hours old.

33. *Temperature Control in Steaming Apparatus.*—The temperature of the steaming apparatus should be determined by a thermometer. A temperature considerably lower than 98° C. may exist even though the water is boiling and condensed vapor is escaping from the outlets. The apparatus must be at the required temperature before placing the pats under test. At high altitudes it may be impossible to reach a temperature of 98° C. at atmospheric pressure, and under these conditions a steaming apparatus in which a moderate pressure may be maintained is required for strict compliance with the specifications.

34. *Observations.*—Unsoundness is usually manifested by a change in volume of the pat which causes distortion, cracking or disintegration. It is important to distinguish between actual unsoundness and shrinkage due to improperly made or exposed pats. Fig. 4, in the standard methods of testing, illustrates the appearance of pats which have failed in the soundness test.

DETERMINATION OF TIME OF SET

(See Sections 46 to 49, inclusive, Specifications C 9 - 21.)

35. *Vicat and Gillmore Needles.*—The specifications permit the use of either the Gillmore needle or Vicat apparatus. Weights and diameters of needles should be checked both before use and frequently thereafter. It is sometimes found that even new Gillmore needles are not standard either as to weight or diameter of needle. It is suggested that the weights and the diameters of the needles should meet the requirements given in the specifications within the following tolerances:

NEEDLE	WEIGHT	DIAMETER
Gillmore (small).........	0.25 lb. ± 2.5 grains	$\frac{1}{12}$ in. ±0.002 in.
Gillmore (large).........	1.00 lb. ±10.0 grains	$\frac{1}{24}$ in. ±0.001 in.
Vicat....................	300 g. ± 0.3 g....	1 cm. ±0.2 mm 1 mm. ±0.02mm.

36. *Method of Mixing.*—The method of mixing and size of batch are fully discussed under the section of Mixing Cement Pastes and Mortars. (See Paragraphs 20–25, inclusive.)

37. *Method of Making Specimens.*—The method of making pats is the same as described in the section on Soundness. (See Paragraph 31.) Somewhat more care is required, however, in forming the time-of-set pats. There should be just enough trowelling to give the proper shape and a smooth surface in forming pats for the Gillmore test. An overtrowelled surface or a rough surface may materially affect the apparent time of set. To flatten the top of the pat the paste should be pressed down with a trowel and the surface smoothed with a single stroke of the trowel. The flat surface of the pat should be parallel to the surface of the glass.

38. *Storage of Specimens.*—Pats should be stored in the moist closet during the determination of time of set. See Paragraph 29 for precautions to be observed in connection with the use of moist cabinets. It is usually necessary to take the pats from the moist closet when a determination is being made. They should be left in the air of the laboratory, however, for just as short a time as possible and returned to the moist closet when the trial test has been made.

39. *Method of Testing.*—The needle must be applied normal to the surface and without shock. This is especially difficult in the case of the Gillmore needle, and great care is necessary. The use of a frame, as illustrated in Fig. 5 of the standard specifications, will assist in this regard, provided the surface of the pat is truly horizontal. In determining the end point, the needle will give a crescent-shaped mark some time after the setting time should have been recorded, if not applied normal to the surface. As long as any marking is noted on the pat when the needle is brought normal to the surface it shall be considered that there has been an "appreciable indentation."

TENSION TESTS

(See Sections 50 to 57, inclusive, Specifications C 9 – 21.)

40. *Briquette Molds.*—Briquette molds must be of the form specified in the standard specifications. (See Fig. 6.) A three-gang mold is the most convenient to handle. Clamps should be of a type which will close the joints in the molds completely so as to insure briquettes of specified cross-section. There should be enough metal in the sides of the molds to prevent the possibility of distortion during use.

41. *Storage Tanks.*—Storage tanks should be made of non-corroding material, and may or may not contain racks for holding the briquettes. The use of some form of temperature control device which will keep the storage water at a temperature within ±3° C. (5° F.) of 21° C. (70° F.) at all times is recommended. Water should either be running continuously or be changed frequently. Storage water should be clean and free from any substance which might possibly injure the briquettes. Specimens containing materials possessing

accelerating or retarding effects should not be introduced into the storage tanks with other specimens.

42. *Testing Machines.*—The type of testing machine should be such as to comply strictly with the requirements of the specifications as regards rate and uniformity of application of the load and other details. The clips should be provided with rollers ½ in. in diameter, with full bearing of 1 in. on the briquette, and mounted so as to permit easy turning during the test. Many laboratories do not observe the latter detail sufficiently, with the result that eccentric strains are produced in the briquette and erratic results reported. Moreover, the distance between the rollers in the clips should not vary appreciably, and the clips should be so designed as to keep the rollers in parallel positions during the test. The bearings of the rollers should be well lubricated. Petrolatum or cup grease will be found quite effective. Grit in the bearings or lack of lubrication may produce undue stresses in a specimen, and often result in a jerky or non-uniform operation of a machine. Long use will sometimes so roughen or deform the rollers that they must be replaced.

43. *Calibration of Testing Machine.*—The testing machine should be calibrated frequently in order to determine its accuracy. Knife bearings should be tightly held in position. Loose knife edges have been found productive of large error. The machine may be calibrated by hanging various dead weights from the upper clips and reading the loads recorded by the weighing mechanism of the machine. Daily balancing of the beam is a good habit to acquire.

44. *Molding of Test Specimens.*—See Paragraphs 20–25, inclusive, on mixing cement mortars. Immediately after mixing the standard mortar in accordance with the methods for mixing cement pastes and mortars, the mold should be placed on an oiled glass plate and filled heaping full without compacting. Then the mortar should be pressed in firmly with the thumbs, applying pressure 12 times to each briquette, at points to include the entire surface. The pressure recommended is such that the application of both the thumbs will register a pressure of 15 lb. Then the mortar should be heaped above the mold and smoothed off with a trowel. The trowel should be drawn over the mold in such a manner as to exert a moderate pressure on the material. An oiled glass plate should then be placed on top of the mold, and the mold with the two glass plates turned over. The top plate should then be removed and the operation of heaping, thumbing and smoothing off repeated. Sand should not be allowed to remain upon the face of the mold to which the plate is to be applied, as it may cause a loss of water between the mold and plate, or too thick specimens may result. The joints of the molds should be tightly closed, in order to insure specimens of correct dimensions. Many operators find that a slightly curved trowel is of great assistance in making briquettes because of the ease with which it can be used to clean off the mold faces, and smooth the mortar within the molds. Only mineral oil should be used in oiling molds and plates.

45. *Storage in Moist Cabinet.*—It is intended that the molds containing the briquettes should be stored so that the upper surfaces of the specimens are freely and uniformly exposed to the atmosphere of the moist cabinet. Molds should not be piled one upon the other in such a way that the glass plates of the upper molds exclude the air from the specimens beneath. The briquettes should be kept in moist cabinets until they have attained the age of 24 hours, when they are to be placed in the storage tanks. If the specimens are removed from the molds before they are 24 hours old, they should be replaced on the shelves of

the cabinet until the expiration of the 24 hours. In no case should they be removed from the molds until they are at least 20 hours old. The removal of briquettes from the molds should be done with care, and with as little disturbance as possible. The specimens are not very hard at the end of 24 hours, and rough treatment may produce injuries which, although not apparent, will nevertheless affect the strengths. It will be found very convenient to mark the numbers of the specimens on the mold plate faces of the briquettes, after the plates have been removed, but while the specimens are still in the molds.

46. *Precautions to be Observed in Testing.*—Specimens must be tested as soon as removed from the storage water. Indications of injuries, defects, or unusual appearances should be noted. When a large number of specimens are to be tested at one time they should be removed from the tanks and placed immediately in a pan of water and kept there until actually tested. Briquettes must be carefully centered in the clips, otherwise large variations in the stresses and consequently in the results are probable. It will be found of great assistance in centering the briquettes if the specimens are inserted in the clips so that the smooth surfaces which have been in contact with the mold plate are turned toward the back of the clips, and placed in contact with the guide bars at the back. Bearing surfaces of the briquettes and of the rollers should be clean, smooth and free from sand or grit.

47. After the specimen is properly centered in the clips, just enough load should be applied by hand before starting the mechanically applied load, to take the play of the machine, and prevent jerky application of the load. Very weak briquettes may not permit taking up the play of the machine, and must be handled carefully. Adjustments of the load control mechanism are often needed, and at least daily trials should be made before tests are commenced, to determine if the load is being applied at the specified rate of 600 lb. per minute. In one type of shot-operated machine, No. 12 lead shot is used with very satisfactory results. A quantity of flake graphite added to the shot hopper will insure a smooth and uniform flow of the shot.

TRAINING OF CEMENT TESTERS

Considerable experience is necessary before an operator is able to secure consistent results in repeated tests on the same cement sample. Therefore, before assuming the responsibility of making tests which involve the acceptance of cement, the operator should be thoroughly trained in all details of the standard tests. The following outline of instruction is suggested:

1. Study thoroughly the Specifications and Manual.

2. Make normal consistency tests on several samples from the same lot of cement (at least 10 samples) until successive tests show the same results within 0.5 per cent of water. This will give practice in mixing and in preparing the sample for the time-of-set test by the Vicat method.

3. Practice making pats for time-of-set tests by the Gillmore method and for soundness tests. (These pats are the same). The operation should be repeated until specimens of the specified dimensions can be secured with a minimum of effort. Not less than 20 to 30 trials should be made.

4. Practice making 1:3 standard sand mortar briquettes until the operation can be carried out smoothly and with a minimum of effort. Check uniformity of operation by weighing the freshly-made briquettes. Proper technique has not

been attained until the operator can make successive groups of 6 briquettes with a variation in weight of less than 20 g. per group.

5. Make fineness determinations on several samples from the same lot of cement (at least ten samples) until successive tests on three samples give results within 1 per cent; for example, residues between 19 and 20 per cent.

6. Make five pats each for time-of-set test by the Vicat and Gillmore methods and determine time-of-set. Between intervals of making observations of time of setting continue practice in the determination of normal consistency; this test is most important since the quantity of mixing water used in the other tests depend on it.

7. Parallel tests on five different days on five samples of cement should be made by the skilled operator and the operator in training. The samples should be preferably from different lots of cement. The tests should include:

Normal consistency;
Time of setting (Vicat and Gillmore methods);
Soundness;
Standard sand briquettes (6 in a batch, 3 each for test at 7 and 28 days);
Fineness.

In the tests for normal consistency, time of setting and fineness, for which the results are obtained as the work proceeds, the difference between the results of the two operators should be not greater than the following:

Normal consistency	0.5 per cent of water
Time of setting	30 minutes
Fineness	1 per cent of fineness

If the operator in training has displayed aptitude in carrying out the tests and his results check those of the skilled operator within the limits stated, he may be considered a skilled operator. However, he should not be permitted to make important tests until the results of the 7 and the 28-day strength tests are obtained and his average results are shown to check those of the experienced operator within 10 per cent. Attention is again called to the advantages to be gained by frequent interchange of samples for test by different laboratories.

CEMENT TESTS FOR RESEARCH PURPOSES

In making mortar tests of cement for research purposes the methods of mixing, molding, storing, etc., described above should be followed. In order to obtain representative results it is not sufficient in research work to test only 3 specimens of a kind made from the same batch. At least 5 and preferably 10 specimens should be made for each mix, consistency, age, curing condition, etc., on different days. For example, if it is desired to test specimens of a given mix and consistency at ages of 7 and 28 days, 3 months and 1 year, 4 specimens may be mixed in a batch, one for test at each of the above ages. Additional batches should be mixed on *different days* so that 5 or 10 independent specimens may be averaged for the final result. By following this procedure, accidental variations in the tests are distributed so that no one set of specimens is unduly affected.

Experience and tests have shown that the briquette strength is not a satisfactory criterion of the concrete-making properties of cements where wide variations in age, consistency, mix or other variables are present. For research purposes, it is suggested that tests be made on *concrete* following the methods described in the Standard Methods of Making Compression Tests of Concrete (Serial Designation: C 39 - 25) of the American Society for Testing Materials.[1]

In research work, reliance should not be placed on single tests for fineness, normal consistency, time of setting, and soundness. The results should be based on at least duplicate and preferably on triplicate tests.

APPENDIX

BRIEF BIBLIOGRAPHY ON PORTLAND CEMENT

By Duff A. Abrams

(April 22, 1925)

Notes

The following bibliography refers primarily to portland cement and does not include the voluminous literature on concrete and reinforced concrete. Detailed bibliographies are contained in many of the papers. Periodical literature should be consulted for recent developments.

More complete reference lists on certain subjects may be found in:

Manufacture of Cement, Raw Materials, Etc.:
  "Cements, Limes and Plasters," by E. C. Eckel (1922).
Concrete and Reinforced Concrete:
  "Concrete Plain and Reinforced," by Taylor and Thompson (1916).
  "Étude Expérimentale du Ciment Armé," by R. Feret (1907).
Concrete in Sea Water:
  "Marine Structures," by Atwood and Johnson (1924).
Concrete Exposed to Alkali:
  Bulletin 1314, U. S. Dept. of Agriculture (1925).

Periodicals

Proceedings, American Society for Testing Materials, Philadelphia.
Transactions, American Society of Civil Engineers, New York City.
Concrete, Detroit (Successor to *Cement Age*).
Engineering News-Record, New York City.
Rock Products, Chicago.
Trade Information Bulletins, U. S. Dept. of Commerce, Government Printing Office.
Tests of Metals, Watertown Arsenal, Government Printing Office.
Reports, Chief of Engineers, U. S. Army, Government Printing Office.
Mineral Resources (1901 to date), U. S. Geologic Survey, Government Printing Office.
  Statistics of manufacture of cement, exports, etc.
Proceedings, International Assn. for Testing Materials (Discontinued **1914**).

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[1] A.S.T.M. Standards Adopted in 1925.

Proceedings, German Portland Cement Manufacturers, Berlin.
Mitteilungen, Materialprüfungsamt zu Berlin, Lichterfelde West.
Zement, Berlin, (Charlottenburg) Germany.
Revue des Materiaux de Construction et de Travaux Publics, Paris.
Ciment, Paris.
Proceedings, Institution of Civil Engineers, London.
Engineering, London.
Pamphlets by British Portland Cement Research Assn., Gravesend, England.

HISTORICAL

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DISCUSSION

Mr. Merriman.

MR. THADDEUS MERRIMAN.[1]—In the Manual proposed by Committee C-1 there are raised many interesting points which have to do with the testing of cement. The first of these is the suggestion that the temperature of the room in which the specimens are prepared be maintained within 10° plus or minus of 70° F. Immediately following is the suggestion that the temperature of the storage water be maintained within 5° plus or minus of 70° F. So far as these two points are concerned, they can, more or less readily, be met. When, however, we come to the moist closet, it appears that the temperature therein should also be held within 5° plus or minus of 70° F. If the moist closet were placed in a room absolutely conditioned to the suggested temperatures, it would still be necessary to have another apparatus for regulating the temperature within the closet. Taken all in all, the suggestions presented in the Manual would, if adopted, result in requiring every laboratory to install much apparatus of doubtful value. The crux of the entire procedure of cement testing lies in four main points as to each of which the Manual makes no suggestion. These points are the following:

1. The quantity of water with which the cement is tempered;
2. The temperature reached by the briquettes while in the moist closet;
3. The loss of water by evaporation while in the moist closet; and
4. The alkalinity of the storage water.

The present specification is built up around the so-called "normal consistency." The quantity of water necessary to produce this consistence depends on so many factors that hardly ever will two operators determine and use the same amount. At the very outset of any series of tests made on the same cement in different laboratories the conditions, therefore, are like those at the start of a handicap race. In a few cases the start is from scratch, while in others the handicaps are many yards.

The phenomena of cement setting are purely chemical. They are not in any sense physical. If two samples of a cement are to be compared they must be mixed with equal quantities of water else the

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[1] Chief Engineer, Board of Water Supply, City of New York.

Mr. Merriman.

results will differ because the kind and quality of the hydration products is dependent on the concentration of the solution in which the reactions occur. More water makes a weaker solution in which a particular type of hydration occurs, while less water results in a stronger solution and in some other type of hydration product. The quantity of water used must be sufficiently large to insure the occurrence of the same kind of hydration in all of the samples.

By mixing samples of several cements with equal and sufficient quantities of water it becomes possible to compare their characteristics, and if the quantity of water employed is approximately the same as that with which the cement is used in actual practice, the results obtained will be representative of those which may be expected in the final concrete or mortar.

The second of the points above noted is of great importance. As the hydration of a cement proceeds, the temperature of the mass rises above that of the surrounding atmosphere and the cement loses water by evaporation. The temperature attained by any mass of cement in setting is dependent on its volume. A single briquette in a moist closet will not become as warm as will the briquette in a moist closet filled to capacity. As the temperature of a cement sample rises the hydrations proceed more rapidly and the evaporation rate is increased.

So it is that no two so-called moist closets will subject the samples within them to the same curing condition unless indeed they stand in the same room and each contains an equal number of cement specimens and all of the same size and similarly placed within the closets. Even so, the samples on the lower shelves will be exposed to different conditions as compared with those on the upper, because the temperature and moisture conditions at the top of a moist closet always differ from those at its bottom.

As the temperature of the specimen rises it gives up moisture to the air of any moist closet irrespective of the relative humidity. Any body warmer than the air in which it is placed will give up moisture to that air. In consequence of this condition all samples of a cement in a moist closet lose water. As between two moist closets, unless the conditions are such as to produce equal losses, the hydration products formed in one case will therefore be different from those formed in another.

The fourth point listed calls attention to the accelerating effect produced on cement hydration by the presence of the carbonates and hydroxides of sodium and potassium in the storage water. These compounds are always present in portland cement, in amounts ranging from 0.25 to 1.25 per cent. They are readily soluble and so pass from

Mr. Merriman. the cement into the water. If running water circulates through the storage tanks they are removed but if the water be stagnant their concentration steadily increases as new samples are introduced. In the extreme case, where the water is seldom renewed but more water is added to make up evaporation losses, the alkali concentration may become very great. The effect of the alkalies is to produce high early strength. It is possible, by storing briquettes in alkaline solutions, to increase the early strengths by from 10 to 40 per cent as compared with storage in normal water. Such storage will often produce a strength at 28 days fully as great as a whole year's water storage.

In addition to the foregoing causes which result in different test results there is still another which is due to the different surface conditions of the grains in different lots of Ottawa sand. These differences may be due to the natural conditions of the sand surfaces or to changes in the condition of these surfaces due to differing air and gas conditions at the several points where they are kept. By standardizing the surfaces of the Ottawa sand grains by washing them clean with hydrochloric acid the early strengths may be substantially increased because of the better adhesion which the cement then develops with the sand surfaces.

The aggregate effect of the foregoing causes of difference may well exceed 100 per cent and so it is not surprising that comparable results are seldom to be obtained on the same cement when tested at different laboratories under the present testing procedure. Committee C-1 has before it a problem of great extent and the development of a standard and uniform test procedure is greatly to be desired.

Committee C-1 in its report recommends the elimination of the specific gravity requirement. This proposal comes as a new one to many of us and the report is silent as to the reasons for the recommendation. It appears to the speaker that the proposed elimination would be most unfortunate because, as he understands the meaning of this test, it is the only one now contained in the specifications that is an index of the degree to which a cement has been calcined. It is to be hoped that the committee will reconsider this matter and that, if the recommendation be again presented to the Society, the committee will set forth the reasons on which its conclusion is based.

Mr. Johnson. MR. N. C. JOHNSON.[1]—I was particularly interested in Mr. Merriman's reference to the presence of the carbonates and hydroxides of sodium and potassium in storage water. I have seen storage tanks that were almost crystallin with calcium and aluminum hydroxides, but the presence of sodium and potassium is, I confess, new to me.

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[1] Member of Firm, Hool and Johnson, New York City.

Mr. Johnson. But it seems also that the practice of using tap water in standard tests is one which should be discountenanced in favor of distilled water. There are so many things that vitally affect the cement reactions that it would be advisable if the standard specifications required the use of distilled water. The quantity is so small that this should prove no hardship. Our coagulated waters and our softened waters often have very unfortunate reactions with cement, and waters containing iron react peculiarly with different cements. This is largely an unknown land, but at the present time we have knowledge of over 2800 substances which will vitally affect the cement reaction, sometimes inhibiting it altogether, sometimes inhibiting it in the absence of carbon dioxide, and sometimes accelerating it with carbon dioxide, which will also play a pronounced part in each reaction. Further information on the effect of sodium and potassium hydroxide would be very interesting. Sodium in particular seems to be very queer in its reactions with cement.

Mr. Merriman. MR. MERRIMAN.—On much concrete work in sheltered situations where rain water does not come on to the surfaces, there is to be found a form of efflorescence which, in nearly every case, is sodium or potassium carbonate. The only source of the sodium or potassium is the cement itself.

Mr. Butterfield. MR. E. E. BUTTERFIELD.[1]—I should like to add the voice of another department of the City of New York, the Office of the President of the Borough of Queens, to what Mr. Merriman has said in relation to the proposed elimination of the specific gravity from the specifications for portland cement. The specific gravity is about the only requirement in the specifications for portland cement which is susceptible to precise physical measurement. From the standpoint of the practical physicist or the analytical chemist there is ground for reasonable doubt about the soundness of principle and the reproducibility of results of the other tests. Let us not discard the one test which fulfills the requirements of a precision physical measurement.

Furthermore I have heard complaints of alleged adulteration of otherwise satisfactory imported portland cements with natural cement. It seems to me that the elimination of the specific gravity opens the door to possible adulteration and changes in manufacturing practice which might or might not be detected by other means.

Mr. Slater. MR. W. A. SLATER.[2]—A short time ago in looking over data, trying to find out what the effect of the strength of cement in compression was on the strength of concrete, I found some data on this

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[1] Chemist, Office of President, Borough of Queens, Long Island City, N. Y.

[2] Engineer-Physicist, U. S. Bureau of Standards.

Mr. Slater. subject in a Bulletin of the Structural Materials Research Laboratory. The strength of the cement was varied by varying its fineness. Cement which, when used in 2 by 4-in. cylinders of 1:3 standard sand mortar, gave a strength of 3200 lb. per sq. in., gave, when used in concrete of varying water-cement ratio, a strength of approximately $S=\frac{14000.}{7^x}$ This is the equation given in Bulletin 1 of the Structural Materials Research Laboratory, to represent the relation between strength, S, and the water-cement ration x. Other cements gave concrete strengths which varied from $\frac{14000}{7^x}$ approximately as the square root of the mortar strength varied from the square root of 3200. The test data are shown in Fig. 1. The ordinates are compressive strengths at 28 days and the abscissas are water-cement ratios. The different characters indicate cements having different mortar strengths. In each diagram the graphs of the equation $S=\sqrt{\frac{K}{3200}}\cdot\frac{14000}{7^x}=\frac{250\sqrt{K}}{7^{\frac{w}{c}}}$ for the highest and the lowest mortar strengths are shown, where K is

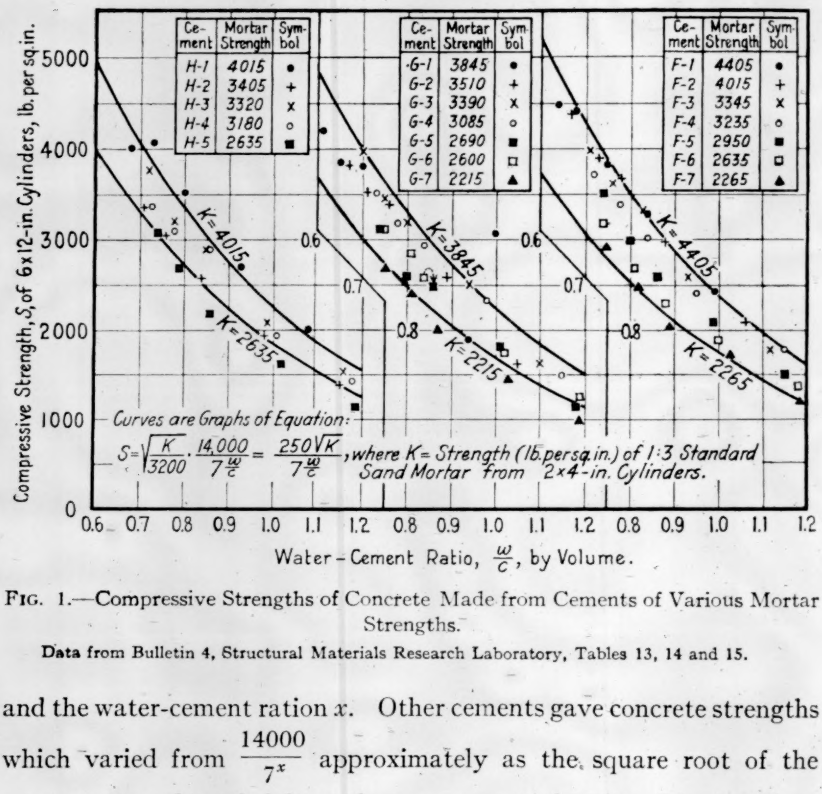

FIG. 1.—Compressive Strengths of Concrete Made from Cements of Various Mortar Strengths.

Data from Bulletin 4, Structural Materials Research Laboratory, Tables 13, 14 and 15.

the compressive strength of 1:3 standard sand mortar tested as 2 by 4-in. cylinders. The agreement of the graphs with the test results is fairly satisfactory. **Mr. Slater.**

It is pretty generally recognized that there is a variation in the strength of concrete with the strength of the cement, even though the cement passes the standard specifications, but I do not recall having seen any expression in the English literature on what that relation is in terms of compressive strength. Prof. Otto Graf of Germany has arrived at an expression similar to this. He uses a water-cement ratio equation which is a little different from the one given here, and in which the strength of concrete is given as proportional to the compressive strength of the cement mortar instead of in proportion to the square root of the compressive strength of cement mortar.

It may be that the compressive strength test is no more reliable as a test of the cement than the tensile strength test, but it is a measure that seems to be more in direct relation to the properties of the concrete which are most used in practice, and therefore should give a better idea of what can be expected of the concrete when the compressive strength of the mortar is known.

REPORT OF COMMITTEE C-3
ON
BRICK

The Conference on Elimination of Unnecessary Sizes and Types of Paving Brick, organized by the U. S. Department of Commerce, has recommended the elimination of one more size, leaving the following four as the recognized sizes and types of paving brick:

	Width, in.	Depth, in.	Length, in.
Plain wire-cut brick (vertical fiber, lugless)	3[a]	4[a]	8½
Plain wire cut brick (vertical fiber, lugless)	3½[a]	4[a]	8½
Repressed lug brick	3½	4	8½
Wire cut lug brick (Dunn)	3½	4	8½

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[a] These bricks are customarily laid in the pavement to give a depth of 3 or 3½ in., respectively, by 4 in. in width and 8½ in. in length.

Committee C-3 recommends that this action of the Conference be approved by the Society and that the Appendix to the Standard Specifications for Paving Brick (C 7 – 15)[1] be amended accordingly. The vacancy on this Conference caused by the death of our distinguished and esteemed member, Will P. Blair, has been filled by appointing T. R. Lawson to represent the committee.

A similar Conference on standard sizes for sand-lime brick has resulted in the promulgation by the Department of Commerce of Simplified Recommendation No. 37. The size recommended in this document is the same as that now called for in the Society's Standard Specifications for Building Brick (C 21 – 20), and Committee C-3 recommends that the above Recommendation be approved by the Society.

A great many tests have been made by some members of the committee to ascertain the difference to be expected in measuring compressive strength when a half brick is tested on edge, as called for in the Standard Specification for Building Brick (C 21 – 20), and when a whole brick is tested flat, as specified in the Tentative Specifications for Concrete Building Brick (C 55 – 24 T). It is recommended that these latter specifications be continued as tentative without revision, in order that the committee may have time to study the test data now available.

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[1] 1924 Book of A.S.T.M. Standards.

Two new methods for measuring the transverse strength of brick are being studied. One of these is an improvement on the apparatus now used for the laboratory test; the other is designed to be used in the field.

The study by the committee of the strength of old brickwork is progressing, but, from the nature of the case, it will be a matter of some years before any definite conclusions can be reported. This work has led to a study of the relation between the properties of individual bricks and the strength of masonry. These matters are more fully covered by a paper on "Brick" by D. K. Boyd presented at this annual meeting of the Society,[1] and in Bureau of Standards *Technologic Paper No. 276* on the "Compressive Strength of Sand-Lime Brick Walls," by H. L. Whittemore and A. H. Stang.

The definition of the word "brick" has been the subject of spirited discussion. Three definitions have been proposed and are now under consideration. This matter is now of practical rather than of academic importance because the committee has been asked to decide whether or not our present Standard Specifications for Building Brick (C 21 – 20) apply to face brick.

During the past year, the committee has added eight new members. Of these, one represents producers of common clay brick, one of cement brick, one of face brick, and two of paving brick. The other three are an architect, an engineer, and a mason contractor.

This report has been submitted to letter ballot of the committee, which consists of 27 members, of whom 22 have voted affirmatively, 1 negatively, and 4 have refrained from voting.

Respectfully submitted on behalf of the committee,

T. R. LAWSON,
Chairman.

W. E. EMLEY,
Secretary.

EDITORIAL NOTE

The proposed revisions of the Standard Specifications for Paving Brick were accepted for publication as tentative and appear on page 907.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II (1925).

REPORT OF COMMITTEE C-7

ON

LIME

Committee C-7 has held two regular meetings since the last annual meeting of the Society.

For convenience, the various activities of Committee C-7 are grouped in order of their respective sub-committees.

Sub-Committee II on Lime for Structural Purposes.—This sub-committee has found through additional research work that the method of controlling the consistency of lime putty as at present specified in the Standard Specification for Hydrated Lime for Structural Purposes (C 6 - 24)[1] is unsatisfactory and submits the following revision for publication as tentative:

Section 12.—Change the second and fifth paragraphs from their present form: namely,

> "The lime shall be made into a stiff putty with water and permitted to soak over night. It shall be molded in a rubber ring such as is used with a Vicat needle, resting the specimen on a glass plate."
>
> "If the penetration is less than standard, the sample may be removed from the mold, mixed with more water, and retested. If the penetration is more than standard, the sample shall be discarded and a new one prepared."

to read respectively as follows:

> "The lime shall be made into a stiff putty with water, stirred vigorously with a trowel or spatula for three minutes and permitted to soak over night. It shall be stirred vigorously with a trowel or spatula for three minutes, molded in a rubber ring such as is used with a Vicat needle, resting the specimen on a glass plate."
>
> "If the penetration is less than standard, the sample may be removed from the mold, mixed with more water, stirred vigorously with a trowel or spatula for three minutes, and retested. If the penetration is more than standard, the sample shall be discarded and a new one prepared."

Data are being collected preparatory to the preparation of a specification for sand for lime plastering. Further test data are being collected in connection with the preparation of specifications for lime block.

Sub-Committee III on Lime for the Chemical Industries.—The following actions relative to specifications have been taken:

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[1] 1924 Book of A.S.T.M. Standards.

It is recommended that the Tentative Specifications for Quicklime and Hydrated Lime for Use in the Cooking of Rags for the Manufacture of Paper (C 45 – 24 T)[1] be advanced to standard.

The Tentative Specifications for Quicklime for Use in the Manufacture of Sulfite Pulp (C 46 – 24 T)[2] are recommended to be revised, and continued as tentative in their proposed revised form:

Section 2.—Change Paragraph (*a*) to read as follows by the omission of the words in brackets:

> "(*a*) Either calcium or magnesium lime may be used, but owing to the greater solubility and reactivity of magnesium bisulfite, high magnesium lime gives the best results. The ratio of MgO to CaO shall be reasonably constant and the lime shall be moderately free from impurities. [The lime shall be of such a physical nature that it will form a rapid settling sludge.]"

Change the footnote to this section to read as follows by deleting the carbon dioxide requirement given in brackets:

> "If it is desired to use the non-volatile basis the figures given above should be changed to the following:

	CALCIUM LIME		MAGNESIUM LIME	
	MAXIMUM	MINIMUM	MAXIMUM	MINIMUM
Calcium oxide, per cent	...	94.3	...	56.5
Magnesium oxide, per cent	2.6	....	...	40.4
Oxides of silicon, iron and aluminum, per cent	3.1	....	3.1	....
[Carbon dioxide, per cent	5.0	....	5.0	....]

The Tentative Specifications for Quicklime for Use in Water Treatment (C 53 – 24 T)[3] are recommended to be revised as follows, and continued as tentative in their revised form:

Section 2.—Change Paragraph (*b*) to read as follows by the addition of the italicized word and the omission of the words in brackets:

> "The standard [composition for] quicklime for use in water treatment shall [be a content of] *contain* 90 per cent of available lime. [Lime containing less than 80 per cent of available lime shall be rejected as being uneconomical to use.] All percentages enumerated herein are based on the sample taken at the point of manufacture."

The Tentative Specifications for Hydrated Lime for Use in Water Treatment (C 54 – 24 T)[4] are recommended to be revised as follows, and continued as tentative in their revised form:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 805 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 141.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 807 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 143.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 809 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 147.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 811 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 149.

Section 2.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> "The standard [composition for] hydrated lime for use in water treatment shall [be a content of 85] *contain 90* per cent of available calcium hydroxide (equivalent to [64.3] *68.1* per cent of calcium oxide). [Lime containing less than 80 per cent of available calcium hydroxide shall be rejected as uneconomical to use.] All percentages enumerated herein are based on the sample taken at the point of manufacture."

Specifications for lime for use in the manufacture of calcium arsenate, sugar, glass and bleaching powder and for use in the absorption of carbon dioxide and in causticizing processes are under the consideration of this sub-committee.

Sub-Committee IV on Agricultural Lime.—This sub-committee is continuing its study of solubility of liming materials, and of the use of lime in crop protection work in cooperation with Sub-Committee III.

Sub-Committee V on Methods of Tests.—This sub-committee has found it necessary to revise the Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime (C 25 - 22 T).[1] It is recommended that they be continued as tentative with the following revisions:

Change the Sub-Title "Determination of Available Lime," to read as follows:

> "Determination of Available Lime in Quicklime and Hydrated Lime by the Modified Scaife Method."

Immediately following the sub-title, add two paragraphs, reading as follows:

> " 'Available lime' in quicklime or hydrated lime is that (or those) constituent (or constituents) which enters (or enter) into the reaction under the conditions of the specified method or process.
>
> "The interpretation of results obtained by the following method shall be restricted by the above definition."

Omit the third paragraph of the notes following this method, reading as follows:

> "Results shall be reported on the non-volatile basis."

This sub-committee has under consideration methods of test for determining the sand-carrying capacity of lime, a uniform method for reporting analyses of lime and lime products and methods for the determination of uncombined calcium oxide in hydrated lime.

Sub-Committee VII on Lime for Highways reports that the experimental work now under way has not reached such a degree of completion as to warrant a formal report at this time.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 715 (1922); also 1924 Book of A.S.T.M. Tentative Standards, p. 188.

Committee C-7 recommends the adoption by the Society of the several recommendations contained in this report and tabulated below with the results of the letter ballot vote of the committee.

Items	Affirmative	Negative	Not Voting
I. TENTATIVE SPECIFICATIONS TO BE ADVANCED TO STANDARD			
1. Quicklime and Hydrated Lime for Use in the Cooking of Rags for the Manufacture of Paper (C 45 - 24 T)	21	2	15
II. REVISION OF STANDARDS			
2. Hydrated Lime for Structural Purposes (C 6 - 24)	25	0	13
III. REVISION OF TENTATIVE STANDARDS			
3. Quicklime and Hydrated Lime for Use in the Manufacture of Sulfite Pulp (C 46 - 24 T)	22	1	15
4. Quicklime for Use in Water Treatment (C 53 - 24 T)	25	2	11
5. Hydrated Lime for Use in Water Treatment (C 54 - 24 T)	23	3	12
6. Chemical Analysis of Limestone, Quicklime and Hydrated Lime (C 25 - 22 T)	25	2	11

This report has been submitted to letter ballot of the committee, which consists of 38 members, of whom 29 have voted affirmatively, 2 negatively, and 7 have refrained from voting.

Respectfully submitted on behalf of the committee,

H. C. BERRY,
Chairman.

R. P. BROWN,
Secretary.

EDITORIAL NOTE

The proposed revisions of the Standard Specifications for Hydrated Lime for Structural Purposes were accepted for publication as tentative and appear on page 907.

The Tentative Specifications for Quicklime and Hydrated Lime for Use in the Cooking of Rags for the Manufacture of Paper were approved at the annual meeting and subsequently adopted by letter ballot of the Society on August 28, 1925, and appear in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

The proposed revision of the Tentative Specifications for Quicklime and Hydrated Lime for Use in the Manufacture of Sulfite Pulp; for Quicklime for Use in Water Treatment; for Hydrated Lime for Use in Water Treatment; and of the Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime were accepted. The tentative standards as thus revised appear on pages 612–630.

REPORT OF COMMITTEE C-8

ON

REFRACTORIES

Committee C-8 has held two general meetings during the year and one special meeting which was devoted exclusively to the discussion of the preliminary reports of the Committee on Industrial Survey.

The committee has no new standards to recommend at the present time but has voted to withdraw the existing Tentative Method of Test for Slagging Action of Refractory Materials (C 17 - 19 T).[1] The committee takes this action as a result of the report of the Sub-Committee on Slagging which has shown that the existing method is not satisfactory in its present form since it appears to be more of a test for porosity than resistance to slagging action. This recommendation has been submitted to letter ballot with the following results: Affirmative 33, negative 1, not voting 14.

The Sub-Committee on Microstructure of the Research Committee under the chairmanship of Mr. A. A. Klein has submitted a preliminary report on a very careful microscopic study of fire clays burned under different conditions. This report represents the results of an investigation extending over the past five years. It is quite complete and correspondingly voluminous, and is now being reviewed with a view to condensing it for publication.

For a number of years, Committee C-8 has felt that before it would be in a position to write specifications for refractory materials it would be necessary that it should have adequate information regarding the conditions under which refractories were used in manufacturing processes. The committee has been studying the use of refractory materials in a number of industries. This work has been done through the cooperation of the Refractories Manufacturers Association and preliminary reports have been written on five industries.

The reports on the Industrial Survey of the Malleable Iron Industry and By-Product Coke Ovens were reviewed and discussed at the special meeting in Pittsburgh and are appended hereto as part of this report. The data given for the malleable iron industry has been largely due to Mr. G. L. Morehead and the data on by-product coke ovens was given by Mr. F. A. Harvey.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part I, p. 620 (1920); also 1924 Book of A.S.T.M. Tentative Standards, p. 205.

It will be noted in both of these reports that tentative classifications have been assigned to the refractories used in the different parts of the furnace construction and it is hoped that with this beginning the committee may look forward to the writing of specifications in the near future.

A number of sub-committees, notably those on Analysis, Abrasion, Slagging and Spalling have work in hand that it is hoped will, within another year, yield results that can be reported to the Society.

The committee is arranging for the preparation, in connection with the Bureau of Standards, of some standard samples of burned refractory materials for standardization of analytical work.

This report has been submitted to letter ballot of the committee, which consists of 48 members, of whom 33 have voted affirmatively, 1 negatively, and 14 have refrained from voting.

Respectfully submitted on behalf of the committee,

W. H. FULWEILER,
Chairman.

F. A. HARVEY,
Secretary.

EDITORIAL NOTE

The withdrawal of the Tentative Method of Test for Slagging Action of Refractory Materials, recommended in the report, was approved.

APPENDIX

REPORT ON INDUSTRIAL SURVEY

MALLEABLE IRON INDUSTRY

The term "malleable iron" in this country, refers to a ferrous product cast from a metal prepared by melting and refining to a desired analysis, a mixture of pig iron, steel and malleable foundry remelt. The mixing and subsequent refining process must be carried to a point that will insure the proper percentage of carbon, silicon, sulfur, manganese and phosphorus in the finished bath.

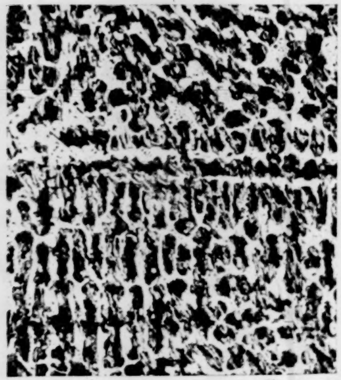


FIG. 1.—Microstructure of Malleable Iron as Cast.

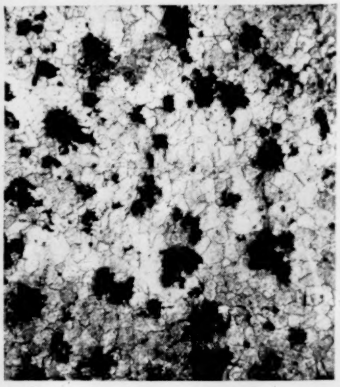


FIG. 2.—Microstucture of Malleable Iron after Heat Treatment

The structure of the product as cast must be such that all of the carbon is combined with the iron to form carbide of iron. The microstructure of the product as cast is shown in Fig. 1. The cast product is subjected to a heat treating process, whereby the chemical union between the carbon and the iron that exists in the product as cast, is broken down and the resulting microstructure is shown in Fig. 2. The irregular black spots in Fig. 2 are nodules of free carbon that has been released from its union with the iron, during the heat treating process, which carbon gathers in the irregular dots and is known as tempercarbon. The product, after passing through the heat treating process, is ductile and malleable and develops a tensile strength of 50,000 lb. or more per sq. in. and an elongation in 2 in. of 10 per cent or more.

The metal for casting may be prepared in a reverberatory air furnace, open-hearth furnace or cupola. The reverberatory air furnace (Fig. 3), which is most extensively used in this country, consists of a firebox, a hearth, separated from the firebox by a bridge wall, a rear bridge wall and two side walls. The roof of the furnace consists of removable bungs or arches, made up by properly fitted refractory shapes, held in metal frames. The reverberatory air furnace in use in this country averages about 6 ft. in width and 18 to 20 ft. in length of hearth. The capacity varies from 5 to 40 tons per charge with an average of around 15 tons per charge. The furnace is charged through openings in the roof, some of the bungs being removed for this purpose, although in a few cases, charging is done through doors in the side walls, the bungs in such cases not being lifted or disturbed. Heat is generated in the furnace by the combustion of coal, oil or gas. Coal is the fuel most commonly used. High temperatures and rapid heating are possible with powdered coal and this method of heating is rapidly coming into favor. The most common method of firing is that of burning bituminous coal on a grate under forced draft. A melting temperature is reached in about 1½ hours after the furnace is lighted, and the metal is completely melted and refined in from 5 to 8 hours. The major portion of the refining takes place during the last 2 to 2½ hours of the heat. Manganese, silicon, and carbon may be removed, first together with iron oxide, forming a slag which readily attacks the side wall bricks along the line corresponding to the top of the bath. A typical air-furnace slag analysis according to H. A. Schwartz in his publication entitled "American Malleable Cast Iron" is as follows:

FeO	28.80	per cent
Fe_2O_3	1.16	"
MnO	4.85	"
SiO_2 (etc.)	50.42	"
Al_2O_3	14.77	"

The slag is skimmed off the top of the bath, either once or twice during the heat. Unless considerable care is exercised during the latter part of the heat, an appreciable quantity of iron will be oxidized, which is not only detrimental to the balance of the metal, but is active in destroying the refractories used in the furnace construction.

The castings when cold, are cleaned, surrounded with packing in iron boxes and submitted to the heat treatment referred to in the first paragraph. The heat treating process is carried out in an oven lined with fire brick and heated from one end by a firebox in which coal is commonly burned. It might be said that some of these ovens in use

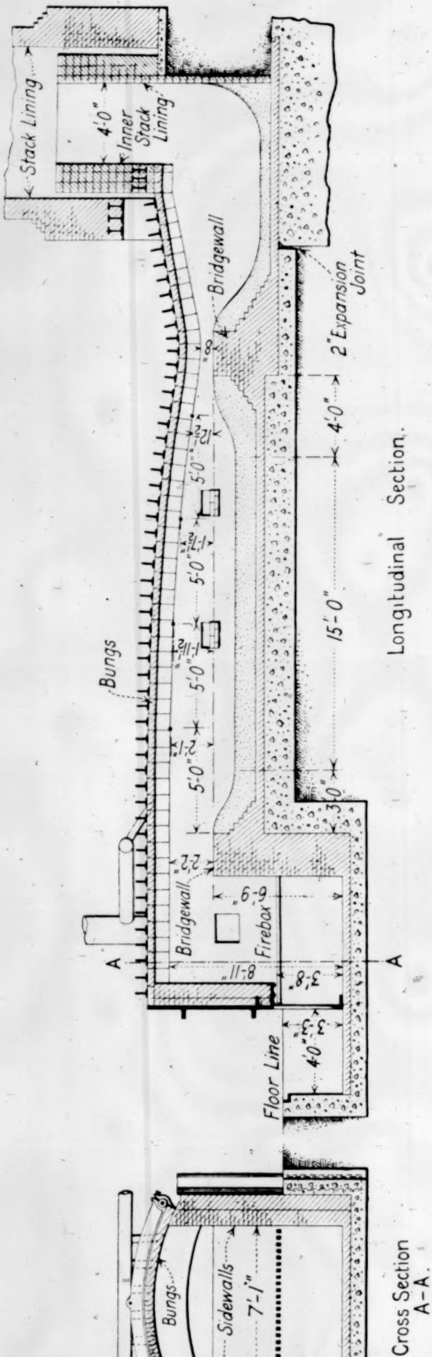

Stack Lining
4'-0"
Inner Stack Lining
Bridgewall
2" Expansion Joint
4'-0"
8"
1'-2½"
5'-0"
1'-1½"
5'-0"
1'-11½"
5'-0"
2'-1"
5'-0"
Bungs
15'-0"
3'-0"
Longitudinal Section.
2'-2"
Bridgewall
6'-9"
Firebox
A
A
8'-11"
3'-8"
3'-3"
4'-0"
Floor Line
Bungs
Sidewalls
7'-1"
Cross Section A-A.

FIG. 3—Reverberatory Air Furnace for Malleable-Cast Iron.

in the malleable iron industry, are heated by powdered coal and oil. The maximum temperature used for heat treating is in the neighborhood of 900° C. The annealing cycle requires on the average of from seven to eight days for completion.

TABLE I.—CLASSIFICATION OF REFRACTORIES FOR MALLEABLE-CAST-IRON INDUSTRY.

	Part (See Fig. 3)	Material Used	Temperature	Spalling	Slagging	Load	Abrasion	Tentative Classification	Remarks
Air Furnace.	Bungs	Fire clay	Up to 1650° C. (3000° F.) near front bridgewall, up to 1370° C. (2500° F.) near center of furnace, up to 1315° C. (2400° F.) at neck	Very important for charging bungs, important to moderate for others	Not of great importance	See note under "Remarks"	Of some importance near front and back bridgewall	H-19	The brick in the bungs are subjected to pressure which may be as high as 1000 lb. per sq. in. due to setting up the clamps too tightly. This would be increased considerably due to thermal expansion when the bungs were heated up.
	Side walls	Fire clay	Varying from 1650° C. (3000° F.) at top of sidewall near the front bridgewall to 1315° C. (2400° F.) near the back bridgewall	Important above the slag line	Important from the hearth up to the lag lines	Maximum of 10 lb. per sq. in.	Unimportant	H-55	
	Fire box	Fire clay	1370° C. (2500° F.) max.	Moderate	Important	Maximum of 10 lb. per sq. in.	Unimportant	H-64	
	Stack lining	Fire clay	1315° C. (2400° F.) max.	Unimportant	Unimportant	Depends upon construction, 50 lb. per sq. in. max. Usually an inner lining is built to a height of 15–20 ft. The load here is not in excess of 15 lb. per sq. in.	Unimportant	H-1	
	Brdg.-walls	Fire clay	1480° C. (2700° F.) max.	Moderate	Important	Unimportant		H-64	
Annealing Ovens	The temperature in the ovens is not over 1700° F., except in the fire boxes, where 2100° F. is the maximum obtained. The load is not in excess of 15 lb. per sq. in., so that the refractory requirements are not at all severe.							M-1	

BY-PRODUCT COKE OVENS

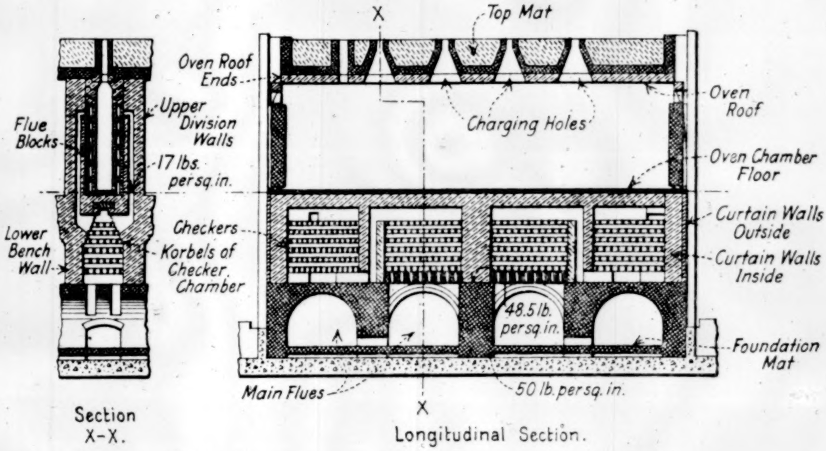
"To make coke, a coking bituminous coal is subjected to heat in a vessel in which the volatile constituents may distill and leave the fixed carbon and concentrated fixed impurities in the form of coke."—HAVARD.

The modern by-product coke oven (Fig. 4) consists of a coking chamber, approximately rectangular in shape, from 30 to 36 ft. in length, 10 to 12 ft. in height, and 16 to 22 in. in width, holding from 10 to 16 tons of coal at a charge. The coking chamber is as nearly gas-tight as possible. It is heated by gas, burned with preheated air in flues at the sides and bottom. The flues may be vertical as in the Koppers ovens or horizontal as in the Semet-Solvay ovens. Usually the gas used is a part of that distilled from the coal. Producer gas, preheated in regenerator chambers, has been used. The coking chamber is charged through holes in the top, from four to six in number, the charge being leveled with a leveling ram. After the coal is coked, doors are raised at the two ends of the oven and an electrically driven pusher ram forces the coke out of the oven into a quenching car. The oven chamber is tapered about 2 in. in 30 ft. to make the pushing

TABLE II.—CLASSIFICATION OF REFRACTORIES FOR BY-PRODUCT COKE OVENS.

Part (See Fig. 4)	Material Used	Maximum Temperature	Slagging	Load as Given for Koppers Ovens	Load Conditions in Semet-Solvay Ovens	Abrasion	Spalling	Tentative Classification
Foundation mat.	Clay	1000° C.	No	25 – 30 lb. per sq. in.	Important	No	No	M-7
Main flues	Silica and clay	540° C. (1000° F.) extraordinary 700 to 760° C. (1300 to 1400° F.)	"	16 – 20 lb. per sq. in.	"	"	"	H-7
Lower bench walls	Silica	1000° C.	"	18 – 20 lb. per sq. in.	"	"	"	H-7
Korbels of checker chambers	Silica	1000 to 1400° C.	"	20 - 25 lb. per sq. in.	"	"	"	H-7
Checkers	Clay and silica	1000 to 1500° C.	"	8 – 10 lb. per sq. in.	No	"	"	H-1
Curtain walls inside	Silica	1000 to 1400° C.	"	......	"	"	"	H-1
Curtain walls outside	Clay	200 to 1000° C.	"	......	"	"	"	L-1
Upper division walls	Silica	1000 to 1400° C.	"	12 – 15 lb. per sq. in.	Important	"	"	H-7
Flue blocks	Silica	1200 to 1500° C.	"	......	"	Important	"	H-9
Oven chamber floor	Clay	1000 to 1200° C.	"	3 – 6 lb. per sq. in.	No	"	"	M-3
Oven roof	Silica	1000 to 1200° C.	"	6 – 10 lb. per sq. in.	Important	No	"	H-7
Oven roof ends	Clay	1000 to 1200° C.	"	5 – 10 lb. per sq. in.	"	"	"	M-7
Charging holes	Clay	800 to 1200° C.	"	......	No	Slight	"	M-1
Jamb blocks	Silica and clay	1000 to 1500° C.	"	15 – 20 lb. per sq. in.	"	Important	Important	H-75
Top mat.	Clay	600 to 1000° C.	"	5 – 10 lb. per sq. in.	"	No	No	L-1

possible and to reduce the abrasive action on the side walls. If by any chance an expanding coal is used, it is very likely to stick and the entire flue structure may be ruined in an effort to push it out. The red hot coke is usually hauled away from the oven block to a separate quenching chamber and quenched by spraying water on it. In some of the older types of ovens the quenching is done right at the ovens. Some steam and spray reach the oven refractories, causing rapid deterioration. The coking process requires from 16 to 30 hours, depending upon the oven width, flue temperatures, thickness of flue walls, and also on the demand for coke and gas. One inch per hour is considered good coking practice, that is, 20 hours on a 20-in. oven, etc. This is frequently exceeded when demand is strong.


X
Top Mat
Oven Roof Ends
Upper Division Walls
Flue Blocks
17 lbs. per sq. in.
Oven Roof
Charging Holes
Oven Chamber Floor
Checkers
Korbels of Checker Chamber
Lower Bench Wall
Curtain Walls Outside
Curtain Walls Inside
48.5 lb. per sq. in.
Foundation Mat
Main Flues
50 lb. per sq. in.
X

Section X-X. Longitudinal Section.

FIG. 4—By-Product Coke Oven

At one end of the coking chamber a riser pipe carries off the gas. This pipe discharges into a "hydraulic main" where the gas is cooled by water sprays, taking out most of the tar and some of the ammonia. The further treatments of the gas and water with recovery of by-products have nothing to do with the refractories.

The oven chamber is sometimes operated under a slight pressure and sometimes a slight vacuum. The condition inside the oven chamber is reducing and in the flues themselves is oxidizing, due to the excess air. Under pressure the flow of gas through the silica brick pores is from the coking chamber to the flues and the oven walls are under reducing conditions; under vacuum the tendency is reversed. This probably has a great effect on the life of the oven but it has seldom been taken into consideration in oven operation, other factors, such as leaky ovens, governing the pressure. The abrasive action which the side walls and bottom blocks must withstand is mentioned above.

There is a very rapid temperature change when the cold, damp coal is dropped into the red-hot oven chamber. The temperature does not, however, drop as low as the critical temperature for crystobalite so that spalling is never a serious factor, except on heating and cooling the entire block in starting and shutting down, and at the ends and charging holes. Fire-clay brick and cast iron are substituted for silica at the ends of the ovens to avoid this spalling. Temperature and load conditions for the various parts of the oven are given in the accompanying table. It should be borne in mind that the charging car with its 15 tons of coal may increase the load very considerably, particularly if there are any irregularities in the charging car tracks.

REPORT OF COMMITTEE C-9
ON
CONCRETE AND CONCRETE AGGREGATES

Committee C-9 has held two meetings since the annual meeting in 1924, and most of its sub-committees have reported more or less progress on their respective problems. In the fall of 1924 a considerable reorganization of the sub-committees was effected, partly to eliminate some of the overlapping that existed prior to that time, thus putting the work on a more logical basis of distribution, and partly to bring about a greater interest in the work through assignment of the members to those sub-committees with whose problems they were directly concerned.

The present status of the committee's activities can best be described by listing the sub-committees as they now exist, together with a statement of their problems and the progress that has been made on these problems.

Sub-Committee I on Definitions.—Terms which appear to need definition are referred to this sub-committee for consideration. Eventually it reports the recommendations of the committee to Committee E-8 on Nomenclature and Definitions. A number of terms are now before this sub-committee for definition, some of which have long been subjects for debate. Among these may be mentioned the terms "workability" and "consistency," which in the opinion of many are different properties, and yet are often used interchangeably. "Density" and "solidity ratio" are again before the sub-committee with an intimation from the committee at large that it does not favor the continued use of the former term as it has been used in the past in concrete parlance. Other concrete terms before the sub-committee, commonly used in different senses, are "disintegration," "accelerator," "admixture," "yield" and "cement content."

Sub-Committee II on Laboratory Tests for Concrete.—This sub-committee combines the functions of a number of the former sub-committees, its duties involving the development of tests to determine the physical properties of concrete. Its first recommendation has been that the Tentative Methods of Making Compression Tests of Concrete (C 39 - 21 T)[1] be advanced to standard. This recommendation has been submitted to letter ballot of the committee with the following results: Affirmative 35, negative 0, not voting 14. While there is some difference of opinion in the minds of the committee in regard to minor details of these methods, the committee believes that the systematic procedure outlined in this tentative standard is worthy of advancement to standard, and so recommends. The sub-committee also presents a bibliography on Methods of Making Flexural and Tensile Tests of Concrete, prepared by the Structural Materials Research Laboratory of the Lewis Institute, which appears in Appendix I to this report.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 579 (1921); also 1924 Book of A.S.T.M. Tentative Standards, p. 211.

Sub-Committee III on Field Tests for Concrete.—The duties of this sub-committee include standardization of methods of making field specimens, including sampling of the concrete when soft, the methods of sampling hardened concrete, and the development of field tests for concrete. This sub-committee has undertaken its work with energy and is investigating the relations that may exist between the strengths of test specimens of different forms and dimensions. It is also producing data to show that in some types of structures the direction of compaction or bedding of the concrete has a marked influence on strength, the latter varying according as the load is applied parallel or perpendicular to the bedding. While further information on these subjects is being sought, the sub-committee has prepared a table of reduction factors for strengths of specimens whose ratio of height to diameter is less than two. The committee has approved the addition of this table to the Tentative Methods of Securing

TABLE I.—COMPRESSIVE STRENGTH OF CONCRETE AS DETERMINED ON SPECIMENS OF VARIOUS SIZE AND SHAPE.

Coarse Aggregate	Slump, in.	Compressive Strength of 1:3:6 Concrete at 28 Days, lb. per sq. in.					
		6 by 12-in. Cylinders	8 by 16-in. Cylinders	4-in. Cubes		6-in. Cubes	
				1[a]	2[a]	1[a]	2[a]
¾-in. gravel	4¼	854	...	1240	952	....	....
¾-in. limestone	4	918	...	1690	1683	....	....
¾-in. gravel	8	799	...	1122	868	....	....
¾-in. limestone	8	742	...	1611	1451	....	....
¾-in. gravel	6	...	754	....	....	1555	1243
1½-in. limestone	6½[b]	...	909	....	....	2206	1735
1½-in. limestone	6[b]	...	1030	....	....	1673	1685
¾-in. gravel	12[b]	...	696	....	....	1092	1069
1½-in. limestone	12[b]	...	800	....	....	1897	1713
1½-in. limestone	12[b]	...	688	....	....	1848	1516

[a] In columns 1 are test results from applying the load parallel to the direction of compaction of the concrete; in columns 2, from applying the load perpendicular to this direction.

[b] Slumps obtained with 8 by 16-in. molds.

Specimens of Hardened Concrete from the Structure (C 42 - 21 T)[1] and accordingly makes the following recommendation:

Add a new Section 6 to read as follows:

"6. In case the dimensions of a specimen are necessarily appreciably different from the recommended shape which requires a height twice the diameter, allowance for the ratio of height to diameter must be made in determining the strength. When the height is more than twice the diameter, that is, when the ratio is greater than 2, no correction need be made. For other ratios corrections should be made as follows:

Ratio of Height of Cylinder to Diameter, $\frac{h}{d}$		
1.75	multiply crushing strength by	0.98
1.50	" " " "	0.95
1.25	" " " "	0.94
1.10	" " " "	0.90
1.00	" " " "	0.85
0.75	" " " "	0.70
0.50	" " " "	0.50

The direction of the application of the load in the compression test shall be stated with reference to the direction of the compaction of the mass of concrete in the structure and where it is practicable the compression load shall be applied in the same direction. In order that the tests be made under uniform conditions as to moisture content, the test specimens shall be completely submerged in water for 48 hours and the compression test shall be made immediately thereafter."

It is recommended that the methods as revised be continued as tentative. The letter ballot vote of the committee on the revision is as follows: Affirmative 32, negative 3, not voting 14.

It is felt that further information is necessary on the relations between the compressive strength of test cylinders made in accordance with the A.S.T.M. standard method and prisms removed from slabs of the same mix, to determine (1) the influence of the difference between the methods of compaction in job concrete and laboratory test cylinders, (2) the practical correction for varying height-diameter ratios of job concrete and laboratory cylinders of the same mix, (3) the influence of the direction in which the compression load is applied with reference to the bedding plane of the coarse aggregate, that is, the values for compressive strength of the concrete in different directions within the same mass of concrete.

The results of a preliminary series of tests on cylinders made in accordance with the A.S.T.M. method and on cubes sawed from slabs made from the same materials as were the cylinders are given in

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 588 (1921); also 1924 Book of A.S.T.M. Tentative Standards, p. 217.

Table I. Cooperation of other laboratories is desired in the matter of securing data and conducting tests:

1. To determine the compressive strength of 4, 6 and 8-in. cubes and of 3 by 6 and 4 by 8-in. prisms sawed from the same mass of concrete;
2. To determine the compressive strength of cored specimens of the same concrete 6 in. in diameter and of varying heights;
3. To determine the compressive strength in different directions within the same mass of concrete;
4. To determine the compressive strength of laboratory test cylinders made by the standard method from the fresh concrete used in the preparation of specimens of the three foregoing items.

The necessity for a method of determining the density of concrete in a structure was discussed by the sub-committee and it was agreed that rather than devise and propose a method for the determination of the density of mass concrete, it would seem more logical to recommend where maximum density of concrete is desired, that the unit weight of air-dry concrete be compared with the maximum unit weight of air-dry concrete obtainable with the same materials in the laboratory.

Sub-Committee IV on Design of Concrete.—This is a new sub-committee, the membership of which has not yet been completed, to be assigned the problem of analyzing the existing data on various theories of proportioning concrete mixtures, and of developing an adequate theory of design. Past experience has indicated that this is the most difficult problem the committee has ever had before it, but a partial solution should be possible when and if the necessary time and effort can be given to the analytical work.

Sub-Committee V on Specifications and Methods of Tests of Aggregates.—This sub-committee embodies two former sub-committees on fine and coarse aggregates, respectively. The new sub-committee has started its work with a definite end in view, namely, to develop standard specifications for aggregates that consist of something more than descriptive matter and grading limits, but nothing sufficiently definite to warrant altering the existing tentative specifications has been accomplished as yet.

Sub-Committee VI on Specifications for Concrete.—This sub-committee will outline general specifications for concrete, probably building on the specifications of the Joint Committee on Specifications for Concrete and Reinforced Concrete as opportunity offers or necessity arises. It will do no research work but will draw upon the other sub-committees for material or other specifications which may be required to complete the general specifications.

Sub-Committee VII on Available Aggregates for Concrete.—This sub-committee has had a somewhat peculiar history. After several years of inactive existence owing to the fact that no one seemed to have any definite idea of its duties, it was discharged in 1923. Immediately thereafter the idea was advanced that a general form for recording data on sources of aggregates would be helpful, and the committee was re-established last year. It immediately went to work along the lines indicated, and has prepared a report form on which data may be entered covering deposits of natural aggregates. This form appears as Appendix II to this report, but the committee would call attention to the need for a more general form, applicable to all materials suitable for concrete aggregates.

Sub-Committee VIII on Deleterious Substances in Concrete.—The duties of this sub-committee involve a study of the various naturally or accidentally occurring materials in concrete which may affect it deleteriously. There is much information on this subject already available which will probably be compiled in the form of a report; there are also definite investigations to be carried out on the effect of such ingredients as coal, mica, lignite and other substances. A general bibliography on the subject is also in course of preparation.

Sub-Committee IX on Admixtures.—This sub-committee will undertake a general study of admixtures which may be added to concrete for the purpose of improving some of its properties. These admixtures are generally, though not necessarily, in the form of finely divided powders. The first work to be undertaken by the committee is therefore the development of a method for determining the weight per unit of volume of such materials under definite conditions of packing. If such a method can be developed, it may prove applicable to many other types of powders and thus serve a general and useful purpose. Its value in connection with concrete is in enabling comparisons to be made on a basis of either weight or volume, since the use of either of these units of measure alone may be misleading, owing to the enormous range in bulking of these materials. A preliminary investigation has already been started in which a number of different methods of packing will be carried out with at least five different materials by twelve different laboratories. In this investigathe cooperation of the producers and of the representatives of the laboratories was freely offered, the materials have been delivered to the participants and the tests are now under way. The results will be available during the coming year. Beyond this the sub-committee has much to do in the study of the effects of admixtures on workability, permeability, durability and other properties of concrete.

Sub-Committee X on Conditions Affecting Durability of Concrete in Structures.—This sub-committee is to make a study of the conditions under which concrete has not been durable. It will emphasize the precautions which must be taken to produce good concrete, whether in methods, materials or workmanship. The sub-committee feels, however, that it should confine itself largely to tests for soundness, durability, etc., without getting too far into the field of structures. Undoubtedly much may be learned from a study of aggregates used in structures which have failed. In this it is cooperating with other sub-committees on the development of accelerated tests of various kinds.

Sub-Committee XI on Elastic Properties of Concrete.—The duties of the sub-committee involve the standardization of methods for the determination of the elastic properties of concrete, including studies of the form of specimens, type of bearing, rate of application of load, sensitivity of measuring devices, etc. It includes also studies of the elastic properties of concretes of different mixtures at different ages under different conditions of moisture, the effect of different aggregates, a determination of the modulus of elasticity of concrete in tension, establishment of Poisson's ratio, and establishment of the effect of brief application of loads including loads applied suddenly or with impact. Plans for the work have been drafted and the sub-committee will soon formulate definite recommendations for conducting tests on concrete specimens of various types.

This report has been submitted to letter ballot of the committee, which consists of 49 members, of whom 36 have voted affirmatively, none negatively, and 13 have refrained from voting.

Respectfully submitted on behalf of the committee,

A. T. GOLDBECK,
Chairman.

J. C. PEARSON,
Secretary.

EDITORIAL NOTE

The Tentative Methods for Making Compression Tests of Concrete were approved at the annual meeting and subsequently adopted by letter ballot of the Society on August 28, 1925, and appear in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

The proposed revisions of the Tentative Methods of Securing Specimens of Hardened Concrete from the Structure were accepted. The tentative methods as thus revised appear on page 631.

APPENDIX I

DIGEST OF LITERATURE ON METHODS OF MAKING FLEXURE AND TENSION TESTS OF CONCRETE

By Duff A. Abrams

(May 1, 1925)

L. J. Vicat, "Treatise on Mortars and Concrete," 1818, English Translation by J. T. Smith, 1837

Apparatus for cantilever load on prisms 4 by 2.5 cm.

Q. A. Gillmore, "Limes, Hydraulic Cements and Mortars," p. 29.

4 by 4 by 8-in. prisms loaded at middle of 4-in. span.

J. Grant, *Proceedings*, Inst. Civil Engrs., 1865-66, 1870-71, Book by Spon, 1875.

Bauschinger, *Proceedings*, Munich Tech. Inst., 1879.

Neat cement to 1:5 mortar on 2.4 by 4.8 (vertical) by 12-in. beam on 10-in. span. Relation between modulus of rupture and tensile strength was 3.93 to 2.46 for the dry and 4.65 to 2.25 for the wet.

H. Fiaja, "Portland Cement for Users" (London), 1884.

1-in. prisms 8 in. long tested on 6-in. span at 7 days loaded at center. Average breaking strength 92.8 lb. See "Portland Cement," by D. B. Butler, 1899, 3rd Ed., 1913.

E. C. Clarke, *Transactions*, Am. Soc. Civil Engrs., Vol. 14, 1885.

Beams 10 in. square and 6 ft. long of natural cement buried in a pit and tested at 6 months. Mixes 1:2:5, 1:3:7, 1:4:9 and 1:6:11; pebbles up to 1 in.

Durand-Claye, *Annales des Ponts et Chaussees,* 1888.

7 specimens ½ in. square, tested on span of 39.4 in. 6 neat cement and 1 of 1:2 mortar; ages of 5 weeks to 6 months.

A. F. Bruce, *Proccedings*, Inst. Civil Engrs., Vol. 113, p. 217, Vol. 118, p. 389. Abstract, *Engineering News*, p. 367, November 9, 1893.

100 tests for modulus of rupture with different kinds and proportions of sand and about 10 tests for resistance to thrust in 10-ft. arch ribs of different concrete mixtures.

Abbott and Morrison, *Engineering News*, December 14, 1893, and May 10, 1894.

Prisms 1 in. square tested on 4-in. span. Ratio of modulus of rupture of neat cement to tensile strength of briquettes was 1.1 to 3.8 Apparatus for testing.

Durand-Claye, Commission des Methods d'Essai, Vol. 4, Sec. B (Paris), 1895.

Flexural tests on 2-cm. square beams of standard and natural sand mortars loaded in middle of 10 to 12-cm. span at 7 and 28 days. Tension tests on briquettes. Mean error for briquettes lower at 7 days, but equal at 28 days.

E. S. Wheeler, Report of Chief Engineer, U. S. Army, 1895, p. 2922. "Materials of Construction," by J. B. Johnson, 1914, 4th Ed., p. 608. "Cement and Concrete," by L. C. Sabin, 1907, p. 331.

10-in. square beams $4\frac{1}{2}$ ft. long of sandstone, limestone and gravel concrete of 1:1.24:3 to 1:6.24:13.80, tested at middle of 4-ft. span; 14, 18, 22 and 30 months, followed by test at later ages on 20-in. span.

R. Feret, "Chimie Applique," by Durand-Claye and Derome, 2nd Ed., p. 419, 1897.

Tests on 3 to 4-in. square mortar specimens 16 cm. long, loaded in middle of 10-cm. span. Specimens of mortar interior with neat cement coating.

W. L. Brown, *Proceedings*, Inst. Civil Engrs. (London), 1898-1899.

Neat and mortar specimens 2 in. deep, 1 in. wide and 30 in. span. 2 brands of cement; coarse sand, poor, fine unwashed sand, and a very coarse sand; center breaking load 5 to 35 lb.

H. Von Schon, *Transactions*, Am. Soc. Civil Engrs., 1899.

Specimen 6 by 6 in. with 18-in. span using 2 brands of cement with boulder stone and sandstone up to $1\frac{1}{2}$ in. as aggregates, of 1:2.4:4 to 1:3.1:5.3 mix with lime as an admixture in one instance. Concludes that no empiric equation can express results.

J. Paterson, *Engineering* (London), August 10, 1900.

Testing cement by modulus of rupture for transverse strain.

R. Moore, *Engineering Record*, February, 1900, p. 187.

Beams 9 in. square, 30 in. long; 27-in. span, 4 brands of cement; neat cement and 1:1 mortar tested at 7 and 28 days. 1:3:6 concrete tested at 8 to $11\frac{1}{2}$ months.

Tests of Metals, Watertown Arsenal, 1900, p. 1110.

1:3:5 concrete prisms 4 to 6 by 6 (deep) by 72 in. tested on spans of 68 and 30 in.; cured 2 days in air, then 2 months in water, then 1 month in air. Hand and machine mixed. $\frac{1}{2}$ to $2\frac{1}{2}$-in. crushed stone aggregate.

Boston Rapid Transit, Annual Reports, 1901–03.

6 by 6-in. beams, 30-in. span, tested at 30 days to 3 years; hand and machine mixed concrete; sand and stone screenings up to $\frac{1}{8}$ in. and trap rock 1 to $2\frac{1}{2}$ in. 1:1.2:1.8 to 1:2.5:4 mixes.

T. S. Clark, *Engineering News*, July 24, 1902, p. 68.

Relation between tensile strength of briquettes and modulus of rupture of 1 by 1 by 8-in. and 3 by 3 by 30-in. beams ranging from neat cement to 1:2:5 concrete.

M. S. Falk, *Transactions*, Am. Soc. Cvil Engrs., 1903.

Specimens 4 by 4-in. of 16 and 36-in. span, loaded at middle at age of $7\frac{1}{2}$ years, with washed gravel aggregate up to $2\frac{1}{2}$ in. 1:2:4, 1:3:5 concrete and 1:2 and 1:3 mortar beams.

E. J. McCaustland, *Transactions*, Am. Soc. Civil Engrs., 1903.

Neat cement beams 2 by $2\frac{3}{4}$ in. deep on 24-in. span loaded at center at age of 1 year.

Tests of Metals, Watertown Arsenal, 1903, p. 531.

Neat cement prisms approximately 4 by 6 (deep) by 24 in. loaded in middle of 21½-in. span at 8 months; cured in air.

A. N. Talbot, *Bulletin 1*, University of Illinois Engineering Experiment Station, 1904.

9 concrete beams; 9 in. wide and 13½ in. deep. Span 5 to 14 ft.; loaded at third points, at 61 to 65 days.

Coelos and Carleton, "Cements, Mortars and Concrete," by Falk.

Concrete beams 6 by 6 in. on 36-in. span, loaded at middle and at two points equi-distant from middle at 121 to 141 days. Tests at Columbia University.

Feret, "Etude Experimentale du Ciment Armé" p. 376, 1905.

Relation between transverse strength and compression studied. Mortar beams 4 by 4 by 16 in.

Sabin, "Cement and Concrete," 1907.

Tensile strength of briquettes, compression tests on 2-in. cubes, and transverse tests on bars 2 by 2 by 8 in. loaded at middle of 5⅓-in. span. Span selected because at this length the modulus of rupture for a 2-in. square specimen is the same as the load. Natural and portland cements used for neat cement and 1:1, 1:2, 1:3, and 1:5 mortars at ages of 7 days to 1 year and water content ranging from 9 to 24 per cent of dry ingredients. Transverse tests on concrete beams 10 in. square by 4½ ft. long, loaded at middle of 4-ft. span.

Humphrey and Jordan, "Portland Cement Mortars and Their Constituent Materials," *Bulletin 331*, U. S. Geological Survey, 1908.

Mortar beams 1 in. square; 13 in. long, loaded at middle of 12-in. span. Values in table are 18 times the breaking load.

R. L. Humphrey, "Strength of Concrete Beams," *Bulletin 344*, U. S. Geological Survey, 1908.

Tests on 108 plain concrete beams 8 by 11 in. and 13 ft. long on span of 12 ft. and 108 tested on spans of 6 to 9 ft. at third points at 4, 13 and 26 weeks. Beams tested on short spans made from larger part of 13-ft. beams after rupture. Concrete of gravel, granite, limestone, and cinder aggregates.

Wm. B. Fuller, *Transactions*, Am. Soc. Civil Engrs., Vol. 59, p. 67, 1907. "Concrete Plain and Reinforced," by Taylor and Thompson, 1916.

Beams 6 by 6 by 72 in., spans 20 and 60 in. at ages of 32 to 35 days using silica sand and crusher-run trap ¼ to 3-in. Mixes ranging from neat cement to 1:6:10. Compression tests made on broken ends of beams. No constant relation between flexural and compressive strength. Tested Little Falls, N. J., 1901.

D. A. Abrams, *Bulletin 71*, University of Illinois Engineering Experiment Station, 1913, p. 24.

Flexural tests on 6 by 8-in. concrete beams loaded at third points of 36-in. span, and compression tests of 6-in. cubes of 1:1:2 to 1:5:10; at about 60 and 100 days.

A. P. Mills, "Materials of Construction," 1915, p. 194.

Beams 8 in. wide by 10 in. deep on spans of 3 to 8 ft. Clean bank sand and crushed rock from ¼ to 1 in. Mixes 1:1½:3 to 1:4:6 tested at 3 months. Tests at Cornell University. Tension tests made on 6-in. square prisms of limestone and sandstone aggregates of 1:2:4 and 1:2½:5 mixes. Modulus of rupture is considerably higher than tensile strength.

D. A. Abrams, "Flexural Strength of Plain Concrete," *Proceedings*, Am. Concrete Inst., 1922. *Bulletin 11*, Structural Materials Research Laboratory, 1922.

750 concrete beams 7 by 10 by 38 in. loaded at third points on 36-in. span. Comparison of flexural and compressive strength for 1:2 to 1:6 mix; relative consistency 0.90 to 1.50; beams tested with top of beam (as molded) up and down; aggregate varying from 0–16 to 0–1½ in.; depth of beam from 4 to 10 in.; slag, limestone, granite and pebbles used as aggregate; cured in dry air, under wet burlap, etc.; tested at 7 days to 1 year; modulus of rupture averaged about 22 per cent of compressive strength.

H. F. Clemmer, "Fatigue of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 408 (1922); see also, *Transactions*, Am. Soc. Civil Engrs., Vol. 87, p. 1180 (1924).

Repeated load tests made on beams 6 in. square, 3 ft. long, of 1:2:3½ and 1:3:5 concrete; beams arranged as cantilevers similar to spokes in a wheel; outer ends loaded by rubber-tired automobile wheel traveling in circular path.

"Tentative Standard Methods of Sampling and Testing Highway Materials," *Bulletin 1216*, U. S. Dept. of Agriculture, May, 1924, p. 26.

Specimens shall be 12 in. wide, 8 in. deep and 30 in. long, tested by center load on 24-in. span.

APPENDIX II

CONCRETE AGGREGATE MATERIAL INVESTIGATION REPORT

.................................., 19......

Investigation of a deposit of.................................has been completed and the
(stone, sand, gravel)
following data obtained: Producing Company:
Owner's Name: ....

Address, ...Location of supply,
....Situated on, ...
(nearest settlement, twp., county) (R. R., canal, river)
or haul to nearest common carrier.................miles over...................................grade
(hauling surface, if any)
.... Sample taken..by
(whether practical for truck haul) (date)
.... Percentage of deposit represented by sample
(person and rank)
is.................... Workable area of deposit is....................with average depth of...............

ft. Stripping would be....................ft., of............................depth of strata or beds
(sand, clay, shale, etc.)
.... Is supply uniform?....................Water supply......................................
(in., ft.) (lake, creek, etc.)
is....................distant. Cost.....................per cu. yd., prepared. Royalty..................
(ft. or fraction of mile)
per cu. yd. Is there any source of contamination known to exist in materials obtained in this section, to be detrimental to their use in concrete; such as natural organic matter or waste from manufacturing plants?..

....

Are there any concrete materials produced commercially in this district?...............

....
(Give largest operations)
Has material from this deposit under investigation been used for concrete construction?........................

If so, list main structures..

Results of tests

Recommendations:

Signed..

Rank..

NOTE.—The above is a suggested form of report to record data on natural deposits of concrete aggregate sources.

A record of this kind on file by Government, state or private engineering corporations would be of great assistance to engineers and contractors.

It is expected that detailed investigation would be made of any material sources to definitely ascertain quality, quantity available and general working conditions before any detailed estimate is completed.

REPORT OF COMMITTEE C-10

ON

HOLLOW MASONRY BUILDING UNITS

Committee C-10 has held two meetings since the last report, when one tentative standard was presented for adoption as standard and three presented for acceptance as tentative. During the year an amendment of one provision of the Tentative Specifications for Load-Bearing Wall Tile (C 34 – 24 T) was suggested and further consideration of the same provision will be had during the coming year. It is therefore not deemed advisable to recommend to the Society any change in the tentative standards at this time.

The results of tests of individual units and masonry, and reports of service obtained during the past year indicate in general the correctness of the requirements of the tentative standards so far as strength and absorption values are concerned. Investigation of service to check the laboratory results with reference to weathering properties has begun and will be continued. Considerable discussion has centered on how the requirements for fire resistance should be expressed. The point has also been raised whether fire resistance is a primary property of the unit or the masonry, since it is generally possible to obtain any desired degree of fire resistance with proper design and construction features for the masonry. The fire tests of load-bearing tile have been continued at the Bureau of Standards and their completion during the coming year will give the necessary information on which to base fire resistance in case it is decided to do so in terms of performance for the different thicknesses of walls or units.

By action of the Executive Committee the scope of the committee was enlarged to include consideration of specifications for masonry units of other materials than burned clay. No work has as yet been undertaken on new projects within the defined scope.

Members of the committee are accredited with eight new Society memberships.

This report has been submitted to letter ballot of the committee, which consists of 19 members, of whom 14 have voted affirmatively, none negatively, and 5 have refrained from voting.

Respectfully submitted on behalf of the committee,

F. J. Huse,
Secretary.

S. H. Ingberg,
Chairman.

REPORT OF COMMITTEE C-11
ON
GYPSUM

Following its established custom, Committee C-11 has had three meetings during the past year. These meetings were held at New York City on October 24, 1924; at Chicago, Ill., on March 25, 1925, and at Atlantic City, N. J., in conjunction with the June, 1925, meeting of the Society.

As in the case of previous years the work for the past year has been productive in the development of information of particular value to the producer and consumer. Any research work that has been necessary has been conducted by the U. S. Bureau of Standards which has supplied information based upon test data. Producer members of the committee have also carried on any necessary check tests dealing with manufacturing processes.

Tentative Specifications for Gypsum (*C 22 - 24 T*).[1]—In 1924, these specifications were amended to include sizes of crushed and ground material. On the recommendation of Sub-Committee I on Gypsum for Various Uses the note following Section 3 is being reworded to agree with similar reference in the recommended standard specifications for gypsum plastering sand. As amended, these tentative specifications are recommended for advancement to standard. The letter ballot of the committee on this recommendation is as follows: Affirmative 29; negative 1; not voting 2.

Standard Specifications for Calcined Gypsum (*C 23 - 22*).—No changes have been made in these specifications other than were recommended in the June, 1924, report of the committee. At that time the recommendation was made that they be submitted to the American Engineering Standards Committee for consideration as American Standard.

Standard Specifications for Gypsum Plasters (*C 28 - 21*).—At the June, 1923, meeting of the Society the committee recommended that these specifications be submitted to the American Engineering Standards Committee for consideration as American Standard.

The committee is now endeavoring to develop specifications for casting, molding, pottery, dental and orthopedic plasters, as well as information on the acoustic properties of plastered panels. The Bureau of Standards has conducted the necessary sound test determinations and will shortly publish a report on the results obtained.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 834 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 175.

Tentative Specifications for Gypsum Plastering Sand (*C 35 – 24 T*).[1]—The committee has continued its efforts to reconcile these specifications as closely as possible to the definition of sand as proposed by Committee E-8 on Nomenclature and Definitions. It has also been thought necessary to provide a material of a composition that will assure a good sand for use with gypsum plasters. The committee approves the recommendation of Sub-Committee II on Gypsum Plasters that the specifications be arranged in two sections, namely, composition and grading. Also, that the provision requiring that "10 per cent by weight be retained on a No. 8 sieve" be changed to "6 per cent by weight," and that, as amended, these tentative specifications be recommended for advancement to standard. The letter ballot of the committee on this recommendation is as follows: Affirmative 29; negative 0; not voting 3. The amended specifications for gypsum plastering sand read as follows:

"1. *Composition.*—Sand used for plastering in which gypsum is employed, shall consist of fine granular material, naturally or artificially produced by the disintegration of rock containing not less than 80 per cent by weight of silica, feldspar, dolomite, magnesite or calcite, and shall be free from saline, alkaline organic or other deleterious substances.

"2. *Grading.*—It shall be graded from fine to coarse, and when dry not more than 6 per cent by weight shall be retained on a No. 8 sieve; not less than 80 per cent by weight shall be retained on a No. 50 sieve; and not more than 6 per cent by weight shall pass a No. 100 sieve. These sieves shall meet the specifications given in the Bureau of Standards' Standard Screen Scale."

Tentative Specifications for Gypsum Wall Board (*C 36 – 24 T*).[2]—Further consideration has been given to the definition, desired tolerance limitations on dimensions and weight, and the rewording of the requirements for strength owing to the adoption of a strength test method by Sub-Committee IV on Testing Methods. These features of the tentative specifications have been rewritten and the specifications, as revised, are appended hereto.[3] Sub-Committee III on Structural Gypsum Products recommends that, as amended, these tentative specifications be referred to the Society with the recommendation that they be advanced to standard. This recommendation is approved by Committee C-11. The letter ballot of the committee on this recommendation is as follows: Affirmative 27; negative 0; not voting 5.

Tentative Specifications for Gypsum Plaster Board (*C 37 – 22 T*).[4]—In the interests of conformity, and for reasons similar to those advanced in the case of the Tentative Specifications for Gypsum Wall Board, the sections of these specifications dealing with the definition, composition and strength have been reworded, and, in the case of weight, the minimum has been changed from 1500 to 1400 lb. These features of the tentative specifications have been re-written and the specifications, as amended, are appended hereto.[1] Sub-Committee III on Structural Gypsum Products recommends that, as amended, these tentative specifications be advanced to standard. This recommendation is approved by Committee C-11. The letter ballot of the committee on this recommendation is as follows: Affirmative 27; negative 0; not voting 5.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 837 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 178.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 838 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 179.

[3] See A.S.T.M. Standards Adopted in 1925.—ED.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 712 (1922); also 1924 Book of A.S.T.M. Tentative Standards, p. 181.

Tentative Specifications for Gypsum Partition and Furring Tile (C 52 - 24 T).[2]—Following the tendency to standardize materials upon a specified performance requirement, additional changes have been made in these specifications. The requirements for core spaces have been simplified and reference to a 2-in. hollow tile has been omitted. The strength test requirement has been reworded owing to the adoption of a strength test method by Sub-Committee IV on Testing Methods, and slight changes have been incorporated in the rate of absorption. These features of the tentative specifications have been re-written and the specifications as revised are appended hereto.[1] On the recommendation of Sub-Committee III on Structural Gypsum Products, it is recommended that, as amended, these tentative specificiations be advanced to standard. The letter ballot of the committee on this recommendation is as follows: Affirmative 25; negative 3; not voting 4.

Standard Methods of Testing Gypsum and Gypsum Products (C 26 - 23).[3]—In compliance with the request of Sub-Committee III on Structural Gypsum Products, Sub-Committee IV on Testing Methods prepared methods for the determination of the compressive and transverse strength and also the absorption of gypsum partition tile or block, and the transverse strength of gypsum boards, with the recommendation that they be added to the present Standard Methods of Testing Gypsum and Gypsum Products. This recommendation is approved by Committee C-11, and the additional methods of testing are appended hereto for publication as tentative. The letter ballot of the committee on the incorporation of these test methods in the present standard methods is as follows: Affirmative 28; negative 1; not voting 3.

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[1] See A.S.T.M. Standards Adopted in 1925.—ED.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 840 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 184.

[3] 1924 Book of A.S.T.M. Standards.

Tentative Definitions of Terms Relating to the Gypsum Industry (C 11 - 24 T).[1]—Sub-Committee V on Nomenclature recommends that the definition of "cement" be amended to include the expression "which becomes plastic upon the addition of water." The revised definition reads as follows:

"*Cement.*—Any material, or a mixture of materials (without aggregate), which becomes plastic upon the addition of water and, when in a plastic state, possesses adhesive and cohesive properties, and hardens in place. The word 'cement' is used without regard to the composition of the material and does not define its use or location of use as do the words 'stucco,' 'plaster' and 'mortar'."

The tentative definition of "gypsum plastering sand" is amended so as to include the material "magnesite" commonly found in sand, and omitting the word "fine" which was considered superfluous when referring to the component parts. The revised definition reads as follows:

"*Gypsum Plastering Sand.*—The fine granular material naturally or artificially produced by the disintegration of rock containing not less than 80 per cent by weight of silica, feldspar, dolomite, magnesite or calcite."

The tentative definition of "mortar" is amended so that the word "masonry" is used in the place of the word "structural." The revised definition reads as follows:

"*Mortar.*—Any material used in a plastic state that can be trowelled, and becomes hard in place, uniting masonry materials. The word 'mortar' is used without regard to the composition of the material, defining its use as a bonding material, as contrasted with the words 'stucco' and 'plaster'."

The sub-committee also recommends the following additional tentative definitions:

"*Gypsum Wall Board.*—A sheet composed of an incombustible core of gypsum, surfaced with paper or other fibrous material which is firmly bonded to the core, and designed to be used, without the addition of plaster, for walls, ceilings or partitions and affording a surface suitable to receive decoration.

"*Gypsum Plaster Board.*—A sheet composed of an incombustible core of gypsum, surfaced with paper or other fibrous material which is firmly bonded to the core, or with intermediate layers of such material within the core, or an incombustible sheet of gypsum with not more than 15 per cent by weight of fiber intimately mixed; and designed to be used as a lath for the reception of gypsum plaster."

The committee recommends that the amended definitions for "cement," "gypsum plastering sand," "mortar," and the new definitions for "gypsum wall board" and "gypsum plaster board" be referred to Committee E-8 for approval and that they be accepted for publication as tentative. The letter ballot of the committee on this recommendation is as follows: Affirmative 28; negative 0; not voting 4.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 849 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 220.

The balance of producer and non-producer members of the committee has been maintained when making the following additions to the membership:

The Higginson Manufacturing Co., Newburgh, N. Y. (Producer.) Representative to be named.

Operative Plasterers and Cement Finishers International Association of United States and Canada, Jersey City, N. J. (Non-Producer.) Represented by Mr. John F. O'Reilly.

Also, in order to facilitate progress and minimize the possibility of duplication of effort, it has been suggested that Sub-Committee V on Nomenclature, and Sub-Committee VI on Fire Resisting Properties of Gypsum Products be dissolved and that in future the work undertaken by these sub-committees shall be assigned to the proper remaining sub-committees.

A paper bearing directly on the work of the committee is being presented this year by Mr. S. H. Ingberg entitled "The Fire Resistance of Gypsum Partitions."[1] This is a compilation of a considerable amount of data from tests that have been made from time to time on the fire resisting properties of gypsum as a partition material.

This report has been submitted to letter ballot of the committee, which consists of 32 members, of whom 31 have voted affirmatively, none negatively, and 1 has refrained from voting.

Respectfully submitted on behalf of the committee,

WARREN E. EMLEY,
Chairman.

VIRGIL G. MARANI,
Secretary.

EDITORIAL NOTE

The Tentative Specifications for Gypsum; for Gypsum Plastering Sand; for Gypsum Wall Board; for Gypsum Plaster Board; and for Gypsum Partition Tile or Block, amended as recommended in the report, were approved at the annual meeting and subsequently adopted by letter ballot of the Society on August 28, 1925, and appear in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

The proposed revisions of the Standard Methods of Testing Gypsum and Gypsum Products were accepted for publication as tentative and appear on page 908.

The proposed revisions of the Tentative Definitions of Terms Relating to the Gypsum Industry were accepted. The tentative definitions as thus revised appear on page 633.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II. (1925).

APPENDIX

PROPOSED REVISIONS OF THE STANDARD METHODS OF TESTING GYPSUM AND GYPSUM PRODUCTS

The following sections are recommended for publication as tentative to be added, when adopted, to the Standard Methods of Testing Gypsum and Gypsum Products (C 26 – 23):[1]

XII. COMPRESSIVE STRENGTH OF GYPSUM PARTITION TILE OR BLOCK

23. (*a*) Not less than ten full-size gypsum tile specimens shall be used.

(*b*) The test specimens shall be dried to constant weight at a temperature not exceeding 110° F. (43° C.), until two successive weighings of the same specimen do not show a variation in excess of 0.5 per cent. At the option of the manufacturer or purchaser, strength tests may be conducted on samples that are dried at room temperature. However, in cases of controversy all test samples shall be dried to constant weight as herein prescribed.

(*c*) The tile or block shall be tested in the position in which they are designed to be used and shall be bedded on and capped with a felt gasket not less than $\frac{1}{8}$ in. nor more than $\frac{1}{4}$ in. in thickness. At the option of the manufacturer or purchaser, or in cases of controversy, the test samples may be suitably bedded and capped with neat gypsum mortar, or the bearing surfaces of the tile may be planed or rubbed smooth and true. When neat gypsum mortar is used for bedding and capping, the test may be conducted after the mortar has set, but not sooner than one hour after the sample has been capped.

(*d*) The loading head shall completely cover the bearing area of the tile, and the applied load shall be transmitted through a spherical bearing block of proper design. The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute.

XIII. TRANSVERSE STRENGTH OF GYPSUM PARTITION TILE OR BLOCK

24. (*a*) Not less than ten full-size gypsum tile specimens shall be used.

(*b*) The test specimens shall be dried to constant weight as prescribed for the determination of the compressive strength.

(*c*) The tile or block shall be supported on its face (flat) on fixed parallel bearings spaced 24 in. (600 mm.) on centers, and shall be loaded through a similar bearing midway between the supports. All bearing and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of $\frac{1}{2}$ in. (12.7 mm.).

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[1] 1924 Book of A.S.T.M. **Standards.**

(*d*) The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute. The modulus of rupture shall be calculated in pounds per square inch from the formula:

$$\text{Modulus of Rupture} = \frac{3wl}{2bd^2}$$

where l = distance between supports in inches;
b = breadth (width) of tile in inches;
d = thickness of tile in inches;
w = load in pounds at failure of test specimen.

XIV. ABSORPTION OF GYPSUM PARTITION TILE OR BLOCK

25. (*a*) The absorption shall be determined upon a test specimen of partition tile or block consisting of approximately one-half of a full-size tile.

(*b*) The test specimen shall be dried to constant weight as prescribed for the determination of the compressive strength.

(*c*) The dried test specimen shall be submerged in water until a constant saturated weight is attained, but in no event for a period of less than two hours, the water temperature being maintained at between 70 and 80° F. (21 and 27° C.). The test specimen shall be removed, the surface wiped off with a damp cloth and the specimen weighed immediately. The percentage of absorption shall be calculated on the dry weight from the formula:

$$\text{Percentage of Abrorption} = \frac{100\,(B-A)}{A}$$

where A = weight of dry test specimen;
B = weight of saturated test specimen.

(*d*) The rate of absorption shall be determined by sealing upon the face of the test specimen, which shall first be dried to constant weight as prescribed for determination of the compressive strength, a glass tube $1\frac{1}{2}$ in. in diameter and 12 in. in length, graduated in centimeters and located centrally over a core. Into this tube shall be poured 250 cc. of water at a temperature of between 70 and 80° F. (21 and 27° C.). The rate of absorption shall be reported in cubic centimeters per minute.

XV. TRANSVERSE STRENGTH OF GYPSUM BOARDS

26. (*a*) Test specimens shall be taken from not less than ten gypsum boards.

(*b*) Strength test specimens shall be 12 in. (300 mm.) in width and approximately 18 in. (460 mm.) in length. They shall be supported on fixed parallel bearings spaced 16 in. (410 mm.) on centers, and shall be loaded through a similar bearing midway between the supports. All bearing and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of $\frac{1}{8}$ in. (3.2 mm.).

(*c*) Test loads shall be applied at a uniform rate of 60 lb. (27.22 kg.) per minute with a permissible variation of ±10 per cent in the rate. Strength test results shall be reported when the load is applied across the fiber of the surfacing and also parallel to the fiber of the surfacing.

DISCUSSION

[Mr. Emley recommended on behalf of Committee C-11 the adoption as standard of the Tentative Specifications for Gypsum Partition Tile or Block (C 52 – 23 T).—Ed.]

Mr. Ingberg. MR. S. H. INGBERG.[1]—The specifications, I believe, are in many ways rather indefinite and it is not clear how some of the requirements are justified. For instance, on rate of absorption, so far as we can see, all the wood-fibered blocks we have will meet that rate of absorption requirement, hence it seems almost a needless complication to require it, and it probably would vary somewhat with the design of the block, whether it is solid or hollow.

Mr. Emley. MR. W. E. EMLEY[2] (*Chairman of Committee C-11*).—In answer to Mr. Ingberg's criticism, we find that the rate of absorption of a gypsum partition tile is one of its most important properties, in that it seems to govern the adhesion of the plaster to that tile. Since gypsum partition tile are designed for erection of non-bearing partitions, the adhesion of the plaster to the tile is one of the most important properties. Adhesion depends on the rate of absorption rather than on the total absorption. Using the method in which this test is conducted, I do not think that whether or not the block has any cores in it will make any particular difference. We have measured the rate of absorption of practically all of the brands of gypsum tile made in the United States, and the figures carried in the specification are based upon the test data so obtained.

Mr. Ingberg. MR. INGBERG.—Have any rate-of-absorption tests been made using an aggregate other than wood fiber? Is it known whether a gypsum-cinder block or gypsum with any of the other aggregates besides wood fiber now in use meets this absorption requirement, and is it known that if it does not it will not form a satisfactory surface for plaster? Those other blocks have been in use for a long time, and unless it is known that this requirement will not unjustly throw them out it does not seem justifiable to include it in the specifications. As I said, the tests indicate that the wood-fibered blocks now made all pass this requirement, and there does not appear to be any object in having it in from this standpoint.

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[1] Physicist, U. S. Bureau of Standards.

[2] Chemist, U. S. Bureau of Standards.

Mr. Emley. MR. EMLEY.—In answering, I should like to ask Mr. Marani how many gypsum-cinder blocks there are on the market for use in the erection of non-bearing partitions.

Mr. Marani. MR. V. G. MARANI[1] (*Secretary of Committee C-11*).—That is almost an entirely local product, manufactured in the East, and represents a very small percentage, taking the total tile manufactured by manufacturers of what we call standard gypsum block, which is a wood fiber block. Gypsum tile manufacturers are in favor of an absorption test because they believe it is a criterion of the adhesion of plaster to that character of base or background. While it is true that the tests made at the Bureau of Standards included all the tile found on the market that are easily available, nevertheless, manufacturers are still experimenting on other types of gypsum tile, and it is for the express purpose of governing those new types of tile that this requirement was put in. That is, we have in mind the manufacture of a very much more porous tile than has been made in the past, and in order to control the question of the adhesion of plaster, we believe, that in a gypsum tile specification, some indication of the adhesive qualities of that tile to plaster should be included. The only way that this feature can be governed is by just such a provision as we have included in these specifications. The evidence of the propriety of this requirement is indicated by the attitude of the manufacturers, who have unanimously voted in favor of it.

[The specifications were then approved for submission to letter ballot of the Society for adoption as standard.—ED.]

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[1] Chief Engineer, The Gypsum Industries, Chicago.

REPORT OF COMMITTEE D-1

ON

PRESERVATIVE COATINGS FOR STRUCTURAL MATERIALS

Committee D-1 on Preservative Coatings for Structural Materials held a meeting in Baltimore on April 10, 1925, at which fifty members were present. The total present membership of the Committee is one hundred and fifty-one members. During the past year, Committee D-1 has been exceptionally active and as a result, a number of standards have been revised, new standards have been recommended and much work along entirely new lines has been carried out.

Some changes in committe organization have been made, the most important of which are the combining of Sub-Committees VII on Accelerated Tests and the Influence of Pigments on Corrosion and XXI on Specifications for the Fundamental Requirements of Paint for Specific Purposes which will in future be known as Sub-Committee VII on Accelerated Tests for Protective Coatings, and the formation of a new sub-committee to be known as Sub-Committee XXV on Cellulose Ester Coatings.

The objects of Sub-Committee VII and Sub-Committee XXI are so closely allied that the members of these sub-committees considered it desirable to join into one sub-committee for the purpose of studying accelerated tests as a means by which to specify paint and varnish materials according to service requirements and not composition. Work has been done during the past four years which amply demonstrates the practicability of accelerated weathering tests and at least a dozen laboratories actively identified with Committee D-1 are now more or less completely equipped for such work. While the fundamental ideas back of the tests now being used by different laboratories are essentially the same, considerable variance in the details of the method of application are already becoming evident, and it is desirable for Committee D-1 to start coordinating this work with the object of bringing about ultimate unanimity in the application of accelerated weathering tests. This is, of course, without the idea of in any way interfering with the development of individual ideas or the research now under way on the fundamentals of the problem.

For many years linseed oil has been bought and sold on a so-called gallon basis but it is usually marketed in large containers, the contents of which are determined by weighing, and the trade has used an empirical weight of 7½ lb. per gal. in calculating the volume. Linseed oil weighs considerably more than 7½ lb. per gal. at all ordinary temperatures and the specific gravity varies with the temperature. Because of the practical difficulty in determining the volume of linseed oil, Committee D-1 has gone on record as approving the suggestion that linseed oil be bought and sold by weight instead of by volume.

The Executive Committee of the Society has requested information concerning the extent of the use of present A.S.T.M. Standards, and, in accordance with this request, the committee has instructed its sub-committee chairmen to furnish the Executive Committee with all available information concerning the use of standards for which Committee D-1 is responsible. In accordance with a request of the Executive Committee of the Society, the committee intends to report in future, research work contemplated or which seems desirable and also information concerning the reasons leading to the proposal of new standards.

Sub-Committee III on Testing of Paint Vehicles has recommended an addition to the Tentative Specifications for Raw Tung Oil (D 12 - 23 T). The addition is to cover Sampling, and Committee D-1 recommends that this addition, as given in the report of Sub-Committee III, be accepted and the specifications as revised continued as tentative.

Sub-Committee VI on Definitions of Terms Used in Paint Specifications has recommended a definition for "screen (sieve)," which Committee D-1 recommends as given in the subcommittee report appended hereto, for publication as tentative to be added when adopted to the Standard Definitions (D 16 - 24).

Sub-Committee VIII on Methods of Analysis of Paint Materials has recommended Tentative Methods of Routine Analysis of White Linseed-Oil Paints. The methods are based on the literature and on experimental work and it is recommended they be accepted for publication as tentative as appended hereto.[1] No criticism having been received of the Tentative Methods of Routine Analysis of Titanium Pigments (D 186 - 24 T), as published in the Proceedings,[2] it is recommended that these methods be advanced to standard.

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[1] See p. 664.—ED.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 859 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 253.

TABLE I.—REPORT ON COLLABORATIVE TESTS ON DETECTION OF SULFUR IN MINERAL SPIRITS.

Analyst	Sample No.	METHOD No. 1 3 hours at 122° F. (A.S.T.M.)	METHOD No. 2 2 hours at 212° F.	METHOD No. 3 $\frac{1}{2}$ hour at Boiling Point of Spirits (Federal Spec. Board)	METHOD No. 4 Copper Strip in Distillation Flask	Remarks
Walker	1	Slightly tarnished.	Considerably tarnished.	Slightly tarnished.	Upper half tarnished, lower half black scale.	Sample No. 1 cannot be classed as a mineral spirit on account of low boiling point, hence Method No. 3 is less severe than should be.
	2	Very slightly tarnished.	Considerably tarnished.	Blackened.	Covered with black scale.	
	3	Practically unaffected.	Very slightly tarnished.	Slightly tarnished.	No scale or black streaks.	
Steele	1	Practically not discolored.	Slightly tarnished.	Considerably tarnished.	Badly blackened, upper part tarnished.	Same as above.
	2	Practically not discolored	Somewhat tarnished.	Completely blackened.	Badly blackened all over.	
	3	Practically not discolored.	Reddish brown color.	Unaffected.	Considerably tarnished.	
Gardner	1	Considerably discolored	Slightly discolored.	Discolored considerably.		
	2	Irregular slightly discolored.	Considerably discolored.	Marked darkening.		
	3	Not discolored.	Very slightly tarnished.	Very slightly tarnished.		
Rather	1	Tarnished.	Light purple.	Steel gray.		Favors Methods No. 2 or No. 3 modified to use a fixed temperature lower than initial boiling point of spirits.
	2	Tarnished.	Heavy purple.	Black sulfide scale.		
	3	Tarnished.	Very slight reddish.	Very slightly tarnished.		
Nelson	1	Tarnished.	Tarnished.	Tarnished to blue finish.	Blackened with black scale.	Methods No. 3 and No. 4 could not be looked on favorably, due to effect of temperature on reaction of sulfur. Prefers Method No. 2.
	2	Tarnished less than Sample No. 1.	Tarnished.	Tarnished much more than Sample No. 1.	Covered with very heavy black scale.	
	3	Not tarnished.	Very slightly tarnished.	Slightly tarnished.	Slightly tarnished.	
Hall	1	Slightly tarnished.	Very slightly discolored.	Slightly tarnished, more than Method No. 1.	Upper half discolored, lower blackened.	Blackening test can be combined with discoloration test, according to last method; otherwise prefer Method No. 3.
	2	Considerably discolored.	Distinctly discolored.	Discolored more than Method No. 1.	Entire strip blackened.	
	3	Not discolored.	Not discolored.	Not discolored.	Slightly tarnished.	
Fuller		Samples not received.				Favors Method No. 3.
Dean	1	Not passing.	Not passing.	Not passing.	Not passing.	Favors Method No. 3.
	2	Passing.	Not passing.	Not passing.	Not passing.	
	3	Passing.	Not passing.	Passing.	Not passing.	
Nemzek	1	Considerably discolored.	Tarnished with precipitate.	Considerably discolored with blackening.	Heavy black deposit.	Method No. 3 preferable, unless method could be developed of heating at lower than boiling point but higher than 212° F.
	2	Slightly tarnished.	Some discoloration.	Marked blackening.	Same as Sample No. 1.	
	3	Untarnished.	Very slightly tarnished.	Very slightly tarnished.	Slightly tarnished.	
Veitch	1	Not tarnished.	Darkened, brownish precipitate.	Darkened, no precipitate.	Blackened, corroded and flaked off.	Method No. 4 most sensitive, but may be too rigid for practical purposes.
	2	Not tarnished.	Darkened more than Sample No. 1.	Darkened more than Sample No. 1, precipitate.	Same as Sample No. 1, but worse.	
	3	Not tarnished.	Tarnished.	Slightly tarnished.	Tarnished.	
Mackenzie	1	Passing.	Not passing.	Not passing.	Not passing.	
	2	Passing.	Not passing.	Not passing.	Not passing.	
	3	Passing.	Passing.	Passing.	Passing.	

TABLE I.—REPORT ON COLLABORATIVE TESTS ON DETECTION OF SULFUR IN MINERAL SPIRITS (*Continued*).

Analyst	Sample No.	METHOD No. 1 3 hours at 122° F. (A.S.T.M.)	METHOD No. 2 2 hours at 212° F.	METHOD No. 3 ½ hour at Boiling Point of Spirits (Federal Spec. Board)	METHOD No. 4 Copper Strip in Distillation Flask	Remarks
Ingalls	1	Appreciable tarnish.	Metal unaffected, but fine precipitate settled to bottom.	Iridescent tarnish and turbidity of hand.	Not carried out because sulfur test is a preliminary to getting distillation range.	Method No. 4 not equivalent to others except for rough classification into good, bad or doubtful spirits.
	2	Slightly less than Sample No. 1:	Positive tarnish of metal.	Badly tarnished and black precipitate.		
	3	Unaffected.	Slightly tarnished.	Slightly uniformly tarnished.		
Giles	1		Slightly tarnished.	Heavily tarnished.	Heavy corrosion.	Federal Specifications Board method probably best, although no corrosion at 212°F. for 2 hours probably satisfactory. Copper strip in flask too severe.
	2		Heavily tarnished.	Very heavily tarnished.	Very heavy corrosion.	
	3		Not changed.	Very slightly tarnished.	Slightly discolored above surface of liquid.	
Richardson	1	Not changed.	Slightly discolored.	Slightly discolored.	Badly blackened by sulfur.	Copper strip in distillation flask offers most possibilities, providing discrimination is made between copper and carbon scale.
	2	Not changed.	Not changed.	Very slightly discolored.	Badly blackened with carbon, which on removal exposed areas blackened by sulfur.	
	3	Not changed.	Not changed.	Not changed.	Slightly tarnished.	
Perdew	1	Not discolored.	Considerably tarnished.	Blackened, no scale.	Upper half tarnished, lower half black scale.	
	2	Not discolored.	Slightly tarnished one side, other side amber color.	Very black, heavy scale.	Covered with scale.	
	3	Not discolored.	Not tarnished.	Very slightly tarnished.	Slightly tarnished, no scale.	

Sub-Committee IX on Varnish has recommended that the Tentative Methods of Testing Oleo-Resinous Varnishes (D 154 - 24 T) be advanced to standard, which recommendation is approved by Committee D-1. Considerable progress in research and testing methods is reported in the report of the sub-committee appended hereto.

Sub-Committee XI on Paint Thinners Other than Turpentine during the past year has investigated the question of the most satisfactory test for the detection of sulfur in mineral spirits. Samples containing varying quantities of sulfur were sent out to the members of the sub-committee for collaborative tests. Four methods for test were suggested:

1. Immersion of a copper strip in the mineral spirits at 50° C. (122° F.) for three hours, as described in the Society's Tentative Method for Detection of Sulfur in Gasoline (D 130 - 22 T).
2. Immersion of a copper strip in the mineral spirits at 100° C. (212° F.) for two hours.

3. Immersion of the copper strip in the mineral spirits at the boiling point of the spirits for one-half hour (Federal Specifications Board method).

4. Placing of copper strip in distillation flask during the period of the distillation.

The sub-committee finds that the first method is not satisfactory for the detection of sulfur in mineral spirits. There also seem to be valid objections against the last method. The opinion on favoring the second or third method is about equally divided. A detailed report on this investigation is given in Table I.

Suggestions have also been received for a modified method whereby the copper strip is immersed in the mineral spirits at a temperature higher than 212° F. but lower than the initial boiling point of the spirits. The sub-committee has not been able to investigate the possibilities of this method and in view of the divided opinion on the other methods no recommendation is made at this time.

On the recommendation of Sub-Committee XII on Turpentine, the following revisions of the Standard Specifications for Turpentine (D 13 - 24)[1] are recommended for publication as tentative:

Title.—Change to read as follows:

> "Specifications for Gum Spirits of Turpentine and for Steam-Distilled Wood Turpentine."

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> "These specifications apply both to the ***spirits of*** turpentine that is distilled from pine oleo-resins, commonly known as 'gum spirits' or 'spirits of turpentine,' and to turpentine commonly known as '***steam-distilled*** wood turpentine' [which is obtained from resinous wood, whether by steam or by destructive distillation]. When ordering under these specifications, the purchaser shall specify whether (a) gum spirits ***of turpentine*** or (b) ***steam-distilled*** wood turpentine is desired. [When wood turpentine is specified, it may be stated whether steam or destructively distilled wood turpentine shall be furnished.]"

Section 6.—Under "Refractive index at 20° C." and under "Residue after Polymerization" change "Wood turpentine" to read "Steam-distilled wood turpentine."

These recommendations are being made because it is felt that the present standard specifications of the A.S.T.M. do not cover and include destructively distilled wood turpentine which, as commercially made, will not comply with these specifications; and to this extent the specifications are misleading and inapplicable.

Sub-Committee XIII on Shellac has prepared Tentative Specifications for Dry Bleached Shellac and Tentative Methods of Testing Shellac Varnish which are recommended as appended hereto[2] for publication as tentative. The present Tentative Method of Test for Determination of Wax in Shellac (D 29 – 24 T)[1] is recommended for advancement to standard, with a slight revision in the note, namely, changing "4 to 5 per cent of wax" to read "4 to 5.5 per cent of wax." The subcommittee is preparing specifications for Orange Shellac.

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[1] 1924 Book of A.S.T.M. Standards.

[2] See pp. 644 and 660, respectively.—ED.

Sub-Committee XIV on the Preparation of Iron and Steel Surfaces for Painting gives in the report appended hereto further progress in the testing of paint on specially prepared steel panels.

Sub-Committee XV on Specifications for Pigments Dry and in Oil When Marketed in That Form has further considered the Tentative Specifications for Iron Oxide and Iron Hydroxide (D 84 – 24 T)[2] and recommends that the last sentence of Section 3 (*a*) be revised to read:

> "The color and color strength, when specified, and the drying time with a vehicle which has been mutually agreed on, shall be equal to those of an agreed sample."

Committee D-1 recommends that the specifications as revised be continued as tentative. After thorough discussion, the sub-committee reports that there is no demand for specifications for a second class of iron mineral pigment consisting of nearly pure iron oxide; also that it is not at present practicable to write specifications for artificial iron oxide pigments.

In 1924 the sub-committee presented proposed specifications for certain pigments in addition to those for which specifications have been before the Society for some years. As there had been but little opportunity for study of these new specifications by the general membership of Committee D-1, they were not approved by the committee for recommendation to the Society as tentative specifications last year, but were reported and published merely as subject-matter for further study.[3] These specifications have been further discussed by the sub-committee and have been revised somewhat. They are now recommended for acceptance for publication as tentative as appended hereto.[4]

Sub-Committee XVIII on Physical Properties of Paint Materials has reported substantial progress in the work of color measurement, its report being appended.

In the report of Sub-Committee XXII on Method of the Application of Paint by Spraying is offered a progress report on the condition of the painting tests at Bridgeville, Pa.

Sub-Committee XXIII on Anti-Fouling Paints has submitted a progress report appended hereto.

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[1] 1924 Book of A.S.T.M. Tentative Standards.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 852 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 246.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, pp. 479–490 (1924).

[4] See pp. 648–659.—Ed.

The recommendations contained in this report have been submitted to letter ballot of the committee and 69 replies have been received. The results of the letter ballot are as follows:

Items	Affirmative	Negative	Not Voting
I. PROPOSED REVISIONS OF TENTATIVE STANDARDS			
Tentative Specification for Raw Tung Oil (D 12 - 23 T)	53	0	16
Tentative Specifications for Iron Oxide and Iron Hydroxide (D 84 - 24 T)	54	0	15
II. PROPOSED NEW TENTATIVE STANDARDS			
Tentative Methods of Routine Analysis of White Linseed-Oil Paints	56	0	13
Tentative Specifications for Dry Bleached Shellac	46	0	23
Tentative Methods of Testing Shellac Varnish	46	0	23
Tentative Specifications for Lithopone	52	1	16
Tentative Specifications for Lampblack	48	3	18
Tentative Specifications for Bone Black	48	3	18
Tentative Specifications for Chrome Yellow	47	3	19
Tentative Specifications for Pure Chrome Green	47	3	19
Tentative Specifications for Reduced Chrome Green	45	0	24
III. TENTATIVE STANDARDS RECOMMENDED FOR ADVANCEMENT			
Tentative Methods of Routine Analysis of Titanium Pigments (D 186 - 24 T)	53	0	16
Tentative Methods of Testing Oleo-Resinous Varnishes (D 154 - 24 T)	51	1	17
Tentative Method of Test for Wax in Shellac (D 29 - 24 T)	46	0	23
IV. PROPOSED REVISIONS OF EXISTING STANDARDS			
Standard Definitions of Terms Relating to Paint Specifications (D 16 - 24)	58	1	10
Standard Specifications for Turpentine (D 13 - 24)	54	0	15

This report has been submitted to letter ballot of the committee, which consists of 151 members, of whom 69 have voted affirmatively, none negatively, and 82 have refrained from voting.

Respectfully submitted on behalf of the committee,

ALLEN ROGERS,
Chairman.

R. L. HALLETT,
Secretary.

EDITORIAL NOTE

The proposed revisions of the Standard Definitions of Terms Relating to Paint Specifications; and of the Standard Specifications for Turpentine were accepted for publication as tentative and appear on pages 917 and 909.

The Tentative Methods of Routine Analysis of Titanium Pigments; of Testing Oleo-Resinous Varnishes; and the Tentative Method of Test for Wax in Shellac were approved at the annual meeting and subsequently adopted by letter ballot of the Society on August 28, 1925, and appear in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

The proposed new Tentative Methods of Routine Analysis of White Linseed-Oil Paints; and of Testing Shellac Varnishes; and the proposed Tentative Specifications for Dry Bleached Shellac; for Lithopone; for Lampblack; for Bone Black; for Chrome Yellow; for Pure Chrome Green; and for Reduced Chrome Green were accepted for publication as tentative and appear on pages 644 and 648–684.

The proposed revisions of the Tentative Specifications for Raw Tung Oil; and for Iron Oxide and Iron Hydroxide were accepted. The tentative specifications as thus revised appear on pages 636 and 646, respectively.

REPORT OF SUB-COMMITTEE III ON TESTING OF PAINT VEHICLES

The sub-committee undertook a series of exposure tests on white paints to determine the relative dirt collection of paints of low and high pigment concentration. The following detailed instructions were transmitted to the members cooperating:

Test Rack.—The test rack is of very simple design, to be constructed of 2 by 4-in. yellow-pine stringers, upon which there should be fastened white pine boards ⅞ in. thick, 1 ft. wide and 10 ft. long, one board for each paint. The boards are to be primed on both sides and edges before fastening to the frame. To save time the priming can be done by the individual members with white lead or with lead and zinc paint reduced to the proper consistency for priming. The priming coat should be allowed to dry outdoors a full week before the first coat of the ready-mixed paints forwarded is applied. The boards are to be marked off into five sections, each 2 ft. long, for the application of the paints of various reductions. The white pine boards should be of carefully selected wood, fairly free from knots, and should be dressed. They should be fastened in place with galvanized screws.

Paints.—Two coats of the ready-mixed paint are to be applied over the priming coat. The paints forwarded consist of the following:

No. 1 Zinc oxide 50 per cent, titanox 50 per cent
No. 2 Zinc oxide 50 " , lithopone 50 "
No. 3 Zinc oxide 30 " , titanox 70 "
No. 4 Zinc oxide 50 " , white lead 50 "

The pigment mixtures were ground in raw linseed oil to a paste containing 80 per cent pigment and 20 per cent linseed oil by weight. These pastes were then reduced to painting consistency by adding 1 gal. of paste to the following liquids in the quantities indicated in gills ($\frac{1}{32}$ gal.).

REDUCTION	RAW LINSEED OIL, GILLS	TURPENTINE, GILLS	LIQUID DRIER, GILLS	HEAT-TREATED TUNG OIL, GILLS
A	14	15	3	..
B	19	10	3	..
C	24	5	3	..
D	18	3¾	2¼	..
E	12	3¾	2¼	4

The tung oil was produced by heating raw tung oil to 275° F. for a period of one and one-half hours.

Two coats of paint are to be applied. The first coat should be applied to the boards that have already been primed, the primed boards being taken indoors and the paint applied and allowed to dry overnight. The boards are then to be placed on the roof for five days for thorough drying. They are then to be taken indoors and the second coat applied and dried overnight. The next day they are to be placed on the roof and fastened into place for permanent exposure. The panels are to be divided off with black lines and neatly numbered.

Although some of the paints may appear slightly heavy, do not thin them. Cross brushing will cause them to flow out to fairly even surfaces. Clean brushes thoroughly before applying each paint.

Exposure.—The test rack should be placed upon the roof of a building where it will get the most severe exposure to dirt and dust collection. The panels should have a southern exposure of 45 deg. to the vertical. The beginning of the exposure period should be not later than January 1, 1925.

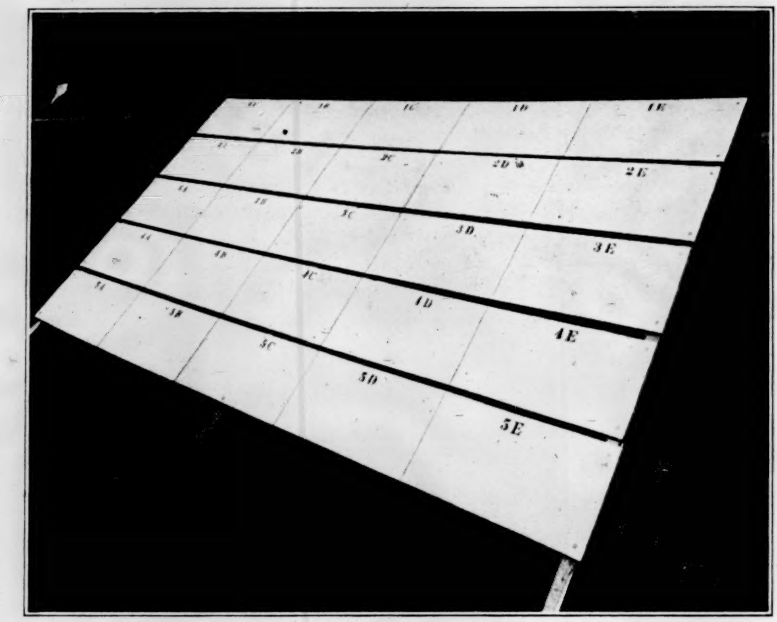

FIG. 1.—Test Rack of White Panels Exposed to Determine Effect of Reduction on Dirt Collecting Properties.

At the request of one member of the sub-committee, white lead was included in the investigation, being assigned No. 5 and reduced in the following manner:

REDUCTION 5*A*	REDUCTION 5*B*	REDUCTION 5*C*
100 lb. paste white lead	100 lb. paste white lead	100 lb. paste white lead
3¼ gal. raw linseed oil	4¼ gal. raw linseed oil	5¼ gal. raw linseed oil
1 pt. drier	1 pt. drier	1 pt. drier
1 pt. turpentine	1 pt. turpentine	1 pt. turpentine

This was used by two members of the sub-committee.

The tests have been made and exposed by G. D. White at Newark, N. J.; R. E. Rogers at Pittsburgh, Pa.; C. R. Cook at Kansas City; F. G. Breyer at Palmerton, Pa.; P. R. Croll at Milwaukee, Wis.; W. D. Jones at San Francisco, Cal.; G. W. Thompson at Sayville, Long Island; and by P. H. Walker and H. A. Gardner at Washington, D. C. There is given below a summary of the results obtained after three months' exposure at the various stations. The results do not vary greatly at the different stations and a photograph of the tests made by H. A. Gardner at Washington, D. C., is reproduced in Fig. 1 to illustrate the condition of the painted panels.

SUMMARY OF RESULTS REPORTED ON APRIL 1, 1925
(After Three Months' Exposure)

P. R. Croll, Milwaukee Exposure. (Tested groups 1, 2, 3, and 4.)
Group 3 cleanest.
Groups 1 and 2 slightly dirtier than Group 3.
Group 4 dirtiest, and uniformly dark.
In Groups 1, 2 and 3, the center panel (*C* reduction) was always darkest; *B* and *D* reductions being cleaner, and *A* and *E* reductions the cleanest. Rain effect upon Groups 1, 2 and 3 caused cleaner surfaces to be shown than on Group 4.

W. D. Jones, W. P. Fuller and Co., San Francisco Exposure. (Tested Groups 1, 2, 3 and 4.)
Group 4 cleanest, reduction 4*E* being cleanest on fence.
All *E* reductions (1*E* to 4*E*) were whiter than others.

George D. White, Newark, N. J., Exposure. (Tested Groups 1, 2 and 4.)
Group 1 whitest.
Group 2 next whitest, and Group 4 darkest.
Reductions *A* and *E* of each group were the cleanest.

P. H. Walker, Washington, D. C., Exposure. (Tested Groups 1, 2, 3, 4 and 5.)
Group 5 most dirt and darkest.
A reduction best of group.
Group 4 next worst but fairly clean and white.
B Reduction best of group.
Group 1 practically the same as Group 4, good.
Reduction *A* best of group.
Group 2, white color, little dirt.
Reduction *A* best of group.
Group 3, whitest, little dirt.
Reduction *A* best of group.

D. L. Rummel, C. R. Cook, Kansas City Exposure. (Tested Groups 1, 2 and 4.)
Group 4 is the darkest group.

H. A. Gardner, Washington, D. C., Exposure. (Tested Groups 1, 2, 3, 4 and 5.)
Group 3 whitest, Group 5 darkest.
In all groups, panels *A* and *E* were cleaner than other panels.

R. E. Rogers, Pittsburgh Exposure.
No report.

F. G. Breyer, Palmerton, Pa., Exposure. (Tested Groups 1, 2 and 4.)
Least dust on 1*E*. Most dust on 4*E*. Dust seems to increase on panels in following order: 1*E*, 1*D*, 1*C*, 1*B*, 1*A*, 2*E*, 2*D*, 2*C*, 2*B*, 2*A*, 4*A*, 4*B*, 4*C*, 4*D* and 4*E*.

The sub-committee recommends that the following requirements for sampling be added to the Tentative Specifications for Raw Tung Oil (D 12 - 23 T)[1] and that they be continued as tentative:

III. SAMPLING

The method of sampling given under Method (*a*) below shall be used whenever it is feasible to apply it. To meet conditions when (*a*) is not applicable, method (*b*) or (*c*) shall be used, according to the special conditions that obtain.

(*a*) *During Loading of Tank Cars or Filling of Containers for Shipment*

The purchaser's inspector shall draw a sample at the discharge pipe where it enters the receiving vessel or vessels. The total sample shall be not less than 1 gal. per each tank car of 10,000 gal. equivalent, and shall be a composite of small samples of not more than 1 pt. each taken at regular intervals during the entire period of loading or filling.

The sample thus obtained shall be thoroughly mixed, and from this composite sample three portions of not less than 1 qt. each shall be placed in clean, dry glass bottles or tin cans, which must be filled with the sample and securely stoppered with new clean corks or well-fitting metal covers or caps. These shall be sealed and labeled distinctly by the inspector, one delivered to the buyer, one to the seller, and the others held for check in case of dispute.

(*b*) *From Loaded Tank Cars or Other Large Vessels*

The total sample shall not be less than 1 gal. per each tank car or 10,000 gal. equivalent, and shall be a composite of numerous small samples of not more than 1 pt. each, taken from the top, bottom, and intermediate points by means of a glass or metal container with removable stopper or top. This device attached to a suitable pole is lowered to various desired depths when the stopper or top is removed and the container allowed to fill. The sample thus obtained shall be handled as in Method (*a*). For large shipments in freighters ranging from 400 to 1000 tons, first draw samples from the top and bottom of both port and starboard sections of the tank. These samples shall be visually examined, and if the general appearance gives indications that the oil is satisfactory, pumping shall be started and 1-pt. samples drawn from a bleeder in the discharge line at least once in every thirty minutes, so that the total mixed sample amounts to as many gallons as there are units of 10,000 gal. in the cargo. These individual samples are composited to make one uniform sample representative of the entire cargo and this is handled as in method (*a*).

(*c*) *Barrels and Drums*

Not less than 10 per cent of the packages in any shipment or delivery of barrels and drums shall be sampled. The packages shall be shaken, rolled, and stirred to thoroughly mix the contents. The samples from the individual containers shall be taken through the bung hole or holes not less than 1¼ in. in diameter bored in the head or side for the purpose. The apparatus for drawing the sample shall consist of a glass tube (known as a thief) about 1 in. in diameter and somewhat longer than the length or diameter of the oil container, a conical stopper that will fit the glass tube and is not more than ½ in. in length, fastened to a stiff metal rod not more than ¼ in. in diameter and not less than 4 in. longer than the glass tube. The stopper is lowered by the rod until it rests on the bottom of the cask, the tube slipped down slowly over the rod, and finally pressed on the stopper. By holding the tube and the rod, the column of oil can then be removed. This process is repeated until the required amount of sample is obtained, which shall be not less than 1 gal. This is mixed and handled as in Method (*a*). During the winter, when the oil is very often in a solid condition, the same procedure should be followed, replacing the thief with a tallow trier, which is essentially a graduated piece of steel tubing semicircular in cross-section and about 3 ft. long. This is pushed into the oil, turned a few times and withdrawn, thereby removing a solid core of oil.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 644 (1923); also 1924 Book of A.S.T.M. Tentative Standards, p. 231.

Respectfully submitted on behalf of the sub-committee,

H. A. GARDNER,
Chairman.

REPORT OF SUB-COMMITTEE VI ON DEFINITIONS OF TERMS USED IN PAINT SPECIFICATIONS

The following is the latest revised recommendation for a definition for "screen" and "sieve," the two terms being synonymous:

> "*Screen (Sieve).*—A plate or sheet or woven cloth with regularly spaced apertures of uniform size, mounted on a suitable frame or holder, for use in separating materials according to size."
>
> "NOTE.—The shape and spacing of apertures, size of wires or threads, thickness of plate or sheet, allowable variations and similar properties should be described in the specifications."

The sub-committee recommends that Committee D-1 approve this definition as a tentative definition to be added when adopted to the present Standard Definitions Relating to Paint Specifications (D 16 – 24).

Respectfully submitted on behalf of the sub-committee,

L. P. NEMZEK,
Chairman.

REPORT OF SUB-COMMITTEE IX ON VARNISH

To facilitate the work of the sub-committee it has been divided into six groups and each group assigned a special problem.

All varnishes used in their work will be recorded according to their physical properties on a carefully prepared data sheet. This gives complete history of the samples and the time of the test. The need of these data has been indicated by changes such as those that have developed in the viscosities of Varnishes A, B, C and D used in the previous work of the committee.

Varnish	Viscosity in Poises at 25° C.	
	April, 1922	September, 1924
A	1.40	0.90
B	1.25	1.30
C	3.40	1.35
D	3.20	1.60

This change in viscosity had taken place in a closed, sealed tube. The extent to which the other properties of these varnishes have been affected is not known.

The reports of the six groups are as follows:

Group 1.—On Effect of Temperature and Humidity upon Drying of Varnish:

J. McE. Sanderson, chairman, reports that the work of designing a suitable machine for the use of this group has been completed. A paper describing this machine will be presented at the annual meeting of the Society.[1] The work of the coming year will be the use of this machine under controlled conditions of temperature and humidity to determine just how these factors affect drying.

Group 2.—On the Use of the Oil Reduction Test for Varnishes of Less than Zero Kauri Reduction:

E. A. Stoppel reports that samples have been sent out covering a range of varnishes of less than Zero Kauri Reduction. The operators have been instructed to determine the non-volatile content of the varnishes, also the non-volatile content after a definite amount of castor oil has been added to the varnish to determine whether the method for determining non-volatile matter should be modified in the case of the less elastic varnishes. These varnishes will then be tested by the Oil Reduction Test which is similar to the test for elasticity or toughness described in the Standard Methods of Testing Oleo-Resinous Varnishes (D 154 – 24) except that heat-bodied linseed oil solution is added to the varnish instead of fused Kauri Gum solution. The effect of linseed oil of several viscosities will be determined.

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[1] J. McE. Sanderson, "A Recording, Drying-Time Meter for Varnishes and Similar Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II (1925).

The results of this test will be compared with cracking of films of various ages when bent over a rod.

These same varnishes will be used by Group 3 for testing the hardness of varnish films and the relation between hardness and oil reduction determined.

Group 3.—On the Hardness of Varnish Films:

L. L. Steele, chairman, reports that this group will determine the accuracy of the swinging beam method of determining hardness in the hands of different operators and the effect of temperature and humidity upon the hardness of a film.

The method which will be used is described in *Circular No. 229,* of the Educational Bureau of the Paint Manufacturers' Association of the U. S.

Group 4.—On Standardization of Service Tests of Exterior Varnishes:

H. A. Gardner, chairman, reports that the subject of exterior exposure tests on varnish was discussed and a series of proposed tests were outlined:

Rack.—Since there is no unanimity of opinion as to the best type of exposure rack, each member of the Group may use such type of rack as he desires, provided the panels face the south at an angle of 45 deg.

Varnishes.—Since the test is to determine the best surface upon which to apply varnishes rather than to determine the durability of different kinds of varnishes, only a relatively few types of varnish will be included in the exposures. These will be manufactured in large quantities in factories and supplied to the Group by three members. They will be of the following types: No. 1 Automobile Finish; No. 2 Ester Tung Oil Spar; No. 3 Linseed Congo Spar.

Panels.—Maple wood panels 6 by 18 by 1 in.; black iron 6 by 12 in. by No. 18 gage; black japanned metal 12 by 4 in. and glass 6 by 12 in.

The exposure tests will be made in three coat work upon wood panels, in two coat work on black iron panels, and in one coat work on the glass panels. The tests on black japanned metal will be in one coat work by dipping, by spraying and by brushing. Three days' drying shall elapse between application of coats and No. 00 sandpaper shall be used lightly for underlying coats. All tests will be run in duplicate.

Any member of the Group desiring may include a similar series of panels in an ultra-violet accelerated testing cabinet if available.

Application.—For brushwork, 2 in. varnish brushes should be used. All applications of varnish whether by brush, spray or by dipping, should be made at a temperature of approximately 70° F. and a humidity of approximately 40 to 50 per cent, if possible, but not greater than 75 per cent.

Time of Exposure.—All panels should be placed on the roof on April 1, 1925.

Inspection.—Inspection shall be made every two weeks, first washing each panel with a sponge and clean water. Loss of luster should be noted. When small cracks develop on a majority of the surface and grow progressively worse, failure should be reported. Other defects as they appear may be noted on the inspection report. Final inspection should be made at the end of a period of nine months unless otherwise instructed.

The subject of a standard black undercoat for testing varnish films was discussed with reference to a report in which it was suggested that an undercoat for all varnishes, whether long oil or short oil, could be made with automobile fender enamel (baked for 45 minutes at 450° F.). The enamel should be rubbed with pumice stone and water. With this undercoat it was believed that one might obtain the maximum life of short oil or long oil varnishes up to the time of the first cracking of the varnish. Although the Group will be furnished with a standard formula for the black undercoat referred to, it is believed advisable in the initial work to furnish each member with panels that have already been treated by one observer with such enamel. If such tests prove satisfactory. it will greatly accelerate the testing of varnishes, since it will be necessary to apply only one coat over a standard surface and expose it rather than wait for several undercoats to dry.

The black japanned panels were furnished by J. E. Booge together with the following formula for the manufacture of the japan.

52 lb. Black Paste	7 lb. Lampblack
	7 lb. Bone Black
	38 lb. Linseed Oil
32 gal. Black Japan	500 lb. Gilsonite
	50 gal. Linseed Oil
	12½ lb. Burnt Umber
	50 gal. Benzine
	100 gal. Kerosine

2 gal. 48° Baumé Petroleum Naphtha.
½ gal. Kerosine.

The Group also discussed the possibility of using glass as a standard surface on which to apply one coat of varnish to determine its durability. The chairman of the Group had been advised by one observer that experiments along this line have not shown entirely satisfactory results because of the fact that varnishes behave in an erratic manner in regard to their adherence to glass, some films peeling more than others. While it is possible that ground glass might overcome this difficulty, the tests at this time will be made upon plain polished window glass. It was also pointed out that glass, being brittle, will be difficult to support rigidly on a test rack. It is possible however, to frame the glass with strips of wood molding and hang the frame from a hook on the exposure rack.

The subject of tin plate as a surface on which to expose varnishes was also discussed, but it was pointed out that the inspection of the films was very difficult over such a surface due to the color of the tin and the underlying scratches on the tin surface which under the microscope might be confused with cracking in the varnish film.

Six 1-pt. cans of each of the varnishes were forwarded to each member of the Group with six black iron panels and eighteen black japanned metal panels. Instructions were given for the preparation of the six glass panels required and the maple-wood panels. Instructions were also given as to the proper method of numbering each set of panels in order to designate the number of coats applied, the type of surface, and the character of varnish used, the designations being 2S-1 to 2S-3 for the black iron panels, G-1 to G-3 for the glass panels, 3M-1 to 3M-3 for the maple panels, BJ-1 to BJ-3 for the hand-brushed japanned panels, SJ-1 to SJ-3 for the sprayed japanned panels and DJ-1 to DJ-3 for the dipped japanned panels.

Group 5.—On the Investigations of Tests for Working Properties:

J. E. Booge reports that among tests the group have been considering have been the gas test, draft test, and the use of an apparatus such as the flowmeter.

Group 6.—On Determination of Acid Value and its Influence in the Thickening with Alkaline Pigments:

H. A. Nelson, chairman, reports that of the two phases assigned to this group for study, namely, (1) acidity in bodied oils and varnishes and (2) the influence of acidity on thickening in enamels, the group is now centering its attention on the first. Eight samples, representing different types of materials, have been agreed upon, as follows:

Two bodied linseed oils:
1. A straight heat-bodied linseed oil; viscosity about 5.0 poises (Tube "S" Gardner-Holdt Viscometer) acid number about 6 to 8;

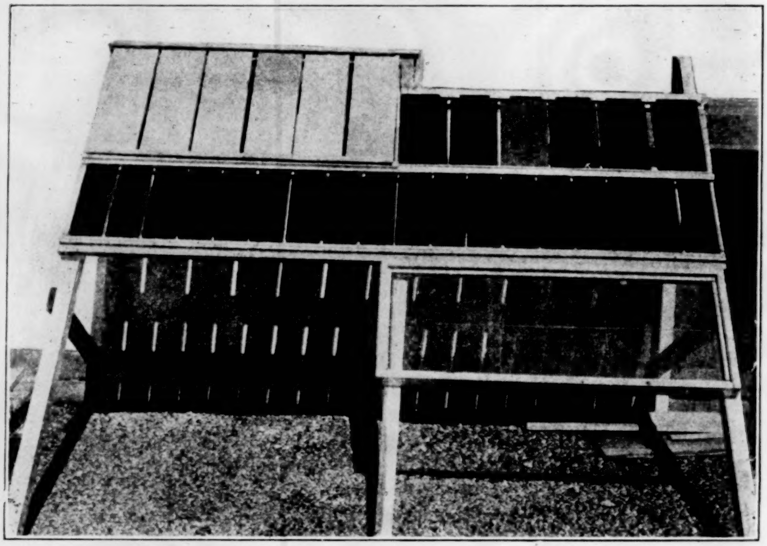

Fig. 1.—View of Test Rack for Testing Varnish.

2. A heat-bodied air-blown linseed oil; viscosity about 7 to 8 poises or about equal to Spencer Kellogg's Diamond "K," acid number about 8 to 9.

Two rosin - tung oil varnishes:
1. With 5 lb. of lime to 100 lb. of rosin
2. With 7 lb. of lime to 100 lb. of rosin

One Damar varnish
One Ester gum - tung oil varnish
One Congo - linseed oil varnish
One cold cut rosin varnish without lime or oil.

The first step contemplated is for each laboratory to run determinations on all samples by the four methods which have been proposed. When this data is assembled, we should be able to size up the essential differences in the methods and their applicability when used by different laboratories on various types of materials.

The sub-committee recommends that the Tentative Methods of Testing Oleo-Resinous Varnishes (D 154 - 24 T)[1] be advanced to standard. The methods referred to include flash point, viscosity and water tests.

Respectfully submitted on behalf of the sub-committee,

E. J. Cole,
Chairman.

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[1] ***Proceedings*****,** Am. Soc. Testing Mats., Vol. 24, Part I, p. 854 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 248.

REPORT OF SUB-COMMITTEE XIV ON PREPARATION OF IRON AND STEEL SURFACES FOR PAINTING

An inspection of the test panels at Altoona, Pa., was made on February 27, 1925, by sub-committee members McDonnell, Sabin, Jennings and Carpenter. The panels were inspected in place on the exposure racks. This was the seventh regular inspection of these panels which, with the exception of panel No. 10 (*a*), had been continuously exposed since February, 1917, a period of 8 years. The results of the inspection are noted in the following:

In the group of new steel panels:

1. Those with surfaces prepared by complete sand-blasting and by pickling and painted very soon thereafter in a warm dry room or in damp or cold atmospheres were still in very good condition.
2. Those with surfaces prepared by sand-blasting and heated for painting; incompletely sand-blasted and painted under normal conditions; sand-blasted or pickled and allowed to rust for 7 days before painting, were in decidedly poor condition.
3. The panels not sand-blasted or pickled were in the poorest condition, and considerably worse than at the time of last year's inspection.

In the group of old steel panels:

4. The panels of old steel were still in fairly good condition.

The individual panels were rated with percentage figures indicating their relative condition based on 100 per cent perfect. The panels are listed below with abbreviated description and the percentage ratings. As the amount of paint applied to the respective panels varied, and as this may be a factor in the results of the test, the average spreading rate per gallon of paint for each panel is also shown:

TESTS ON NEW STEEL, ALTOONA, CONDITION RATINGS OF FEBRUARY 27, 1925

PANEL	DESCRIPTION	SPREADING RATE,[a] SQ. FT. PER GAL.	CONDITION RATING, PER CENT
No. 1	Clean surface, mill scale intact, painted in warm dry room	916	20
No. 2	Mill scale broken, some rust, hand cleaned, painted in warm dry room	826	40

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[a] Average per coat.

PANEL	DESCRIPTION	SPREADING RATE[a] SQ. FT. PER GAL.	CONDITION RATING, PER CENT
No. 10(*a*)	Former Brooklyn panel No. 10 re-prepared by sand-blasting one-half, hand cleaning other half, painted June 13, 1921.		
	Sand blast side	Not recorded	95
	Hand cleaned side	" "	75
No. 3	Sand-blasted, painted immediately after in a warm dry room	785	85
No. 4	Sand-blasted, heated in oven to 225° F. and then painted	899	65
No. 5	Sand-blasted, placed in cool damp room 40 min. and then painted	706	87
No. 6	Sand-blasted, placed outdoors and painted there at a temperature of 25 to 27° F	542	85
No. 7	Sand-blasted, exposed out of doors for 7 days, painted over rust in warm dry room	914	70
No. 16	Incompletely sand-blasted, painted in warm dry room	818	60
No. 8	Pickled, painted in warm dry room	808	87
No. 9	Pickled, exposed out of doors for 7 days, painted over rust in warm dry room	1028	70
No. 11	Pickled, painted in warm dry room	801	90

TESTS ON OLD STEEL, ALTOONA, CONDITION RATINGS OF FEBRUARY 27, 1925.

PANEL	DESCRIPTION	SPREADING RATE[a] SQ. FT. PER GAL.	CONDITION RATING, PER CENT
No. 12	Cleaned with wire brush only	629	85
No. 13	Cleaned with benzine, scraper and wire brush	726	70
No. 14	Benzine applied over rust spots and burned off with torch, then cleaned with scraper and wire brush	730	80
No. 15	Sand-blasted	767	75

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[a] Average per coat.

The tests seem to indicate that the most rapid deterioration of paint occurs where applied over a badly rusted surface. The Altoona tests on new steel indicate that painting will preserve sand-blasted or pickled surfaces longer than hand-cleaned surfaces.

The tests on the old panels at both Altoona and Brooklyn both seem to indicate that there is little advantage in the application of so thorough a method of cleaning as sand-blasting; whereas the test on panel No. 10 (*a*) at Altoona (former Brooklyn panel No. 10, re-prepared by sand-blasting one-half and hand cleaning the other half of one surface) indicates a decided superiority in the sand-blasting method. It would appear, therefore, that there must be some intermediate point in surface condition between that of the old steel panels of the original tests, and that of Altoona panel No. 10 (*a*) which would determine the method required for the best results.

It has been suggested that several panels of varying degrees of deterioration be re-prepared in the same manner as Altoona panel No. 10 (*a*) and observations continued.

Mr. McDonnell has very kindly offered to re-prepare three panels in the manner suggested. The selection of these panels is now under consideration.

There are two of the original Brooklyn panels still exposed. These will be examined at an early date and reported upon later.

The complete test data will be found in the Proceedings of the Society, Vol. XVII, Part I, p. 451 (1917).

A résumé of the test data and results to 1921 will be found in the Proceedings of the Society, Vol. 21, pp. 345–347 (1921).

Respectfully submitted on behalf of the sub-committee,

A. W. CARPENTER,
Chairman.

REPORT OF SUB-COMMITTEE XVIII ON PHYSICAL PROPERTIES OF PAINT MATERIALS

The work of the sub-committee on color measurement has been carried forward with uninterrupted vigor and continuity. After more than three years of arduous preliminary work, and the accumulation of a mass of results, a formal meeting of the sub-committee was held in Brooklyn on September 15, 1924, at which nearly all the members were present or represented, together with associated workers interested in the work, notably Mr. Keuffel, who has given untiring assistance, and the Bureau of Standards, which has been active in this work from the beginning.

Early in the study of the subject the conclusion was reached that spectro-photometric measurement offered the most readily available means for expressing color stimulus in definite physical terms, and while no objection is offered to the use of secondary standards for ordinary practical color matching, the employment of color systems involving arbitrary standards is not feasible for the purposes of color specification. Accordingly most of the several thousand readings accumulated have been made on the spectro-photometer. Much other relevant matter has been considered, but until definite and concordant results with some available type of this instrument can be shown, or the conclusion that the method presents obstacles too difficult for practical purposes has been reached, other lines of investigation must be held more or less in abeyance.

By the able and very extended labors of Mr. Paul all these accumulated data were tabulated, compiled, and plotted so as to be in shape suitable for discussion. After consideration and discussion it was decided to send out another set of paint samples carefully prepared so that, as far as pains-taking care could accompolish it, each item in a set should be identical with the corresponding item in every other set. Further, owing to changes liable to occur with age, all sets were kept under similar conditions and all measurements made at the same time. Five Keuffel and Esser color analyzers were available for this work, and Mr. Keuffel kindly agreed to send out special instructions and means for adjustment of these instruments to the end that they should read as nearly true as possible. This program has been consistently carried out and the results have been recorded in the form of both tables and plotted curves.

A cursory glance at the tables seems to indicate a discouraging lack of concordance, but a more careful analysis shows that three of the observers agree very well, while two in general have a tendency to be low, and, in the case of the other three, information that has come to light since the work was done leads to the conclusion that their results could not be expected to agree with the others very closely. Remembering that a difference of two per cent in the regions of high visibility and up to five or more in the regions of low visibility need not be disconcerting, a survey of the plotted curves shows a very fair agreement in the sets of observations selected as being nearest to normal, except in the case of the whites. Just why this failure should crop us so unexpectedly here is not clear, but it is improbable that there is any fundamental obstacle in the whites greater than is present in other colors.

Respectfully submitted on behalf of the sub-committee,

F. P. INGALLS,
Chairman.

REPORT OF SUB-COMMITTEE XXII ON METHOD OF APPLICATION OF PAINT BY SPRAYING

REPORT OF INSPECTION, OCTOBER 3, 1924, ON TESTS OF SPRAY AND BRUSH APPLICATION OF PAINTS AT BRIDGEVILLE, PA.

On October 3, 1924 an inspection was made of the dwelling houses painted on October 13, 1921. Full particulars concerning this test will be found in the report of the sub-committee for 1922.[1]

A review of the conclusions given in the report will show the situation with respect to the paints after the work was completed. A careful examination of the general appearance of the paints at this time called forth the general conclusion that the jobs are all relatively in the same condition to-day as they were when first completed.

The following are observations made on each house:

House No. 1 (Brush, 3 Non-Union Men).—Chalking moderately, but in good condition. White trim chalking more than gray body.

House No. 2 (Matthews Spray).—There was a slight skin drying, the chalking and condition and trim were the same as in No. 1. The indications were that brushing distributed paint more evenly over the rough surface in No. 1 than spraying. There was a slightly greater discoloration around the nail heads than in No. 1.

House No. 3 (Brush, Union Men).—Chalking the same as No. 1 and No. 2. The surface on this house had been better prepared than the others. This job looks the best of the four and shows that more paint was used on it than on the other three.

House No. 4 (DeVilbiss Spray).—The surface was not well prepared and lack of preparation was apparent. There was uneven distribution of paint, but the condition on heavier coated portions was equal to the others. The chalking was about the same as on the others. On this house, less paint was used than on any of the others.

The conclusion from examination and from their appearance at a little distance would show the relative standing of the houses as follows:

No. 3, No. 1, No. 2, No. 4.

At the time of this inspection the paints applied by the spray method on surfaces of this kind appear to be equally as durable as the paints applied by the hand brush.

Respectfully submitted on behalf of the sub-committee,

R. E. ROGERS,
Chairman.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 384 (1922).

REPORT OF SUB-COMMITTEE XXIII ON ANTI-FOULING PAINTS

Three reports have been previously submitted by this sub-committee: the first, a general series of panel tests covering three vehicles and eight toxic pigments; the second, specific panel tests enlarging upon the best of the first results; and third, applying the most satisfactory results obtained to actual ship-bottoms in regular service. In all these, the toxic pigments were the main point of attack.

In this, the fourth report, is given a series of panel tests carried through during the summer of 1924 upon the more promising anti-fouling paint vehicles, a subject so far partially neglected. The same toxic ingredients were used, these purposely including a variety of toxics, so as to be as fair as possible to any possible vehicle used. One special paint was also included on the panels submerged. The outline below is self-explanatory. Missing paint numbers represent vehicles that were tried and found unsatisfactory during manufacture.

DETAILS AS TO CONSTITUENTS OF PAINTS

(Per cent of whole paint given, unless otherwise stated)

BASE PIGMENT FOR ANTI-CORROSIVE PAINTS:

Zinc dust	3 per cent
Zinc oxide	17 "
Spanish red oxide	10 "
Asbestine	10 "

TOXIC PIGMENT FOR ANTI-FOULING PAINTS:

Copper oxide, electrolytic	5 per cent
Mercury oxide, yellow	1 "
Copper cyanide	5 "
Copper acete-arsenite	5 "

BASE PIGMENT FOR ANTI-FOULING PAINTS (to form of total paint—16 per cent):

Zinc oxide	24.3 per cent
Spanish red oxide	57.1 "
Asbestine	18.6 "
Total	100.00 "

VEHICLES FOR THE PAINTS:

The same vehicle in both anti-corrosive and anti-fouling, unless otherwise specified.

Paint No. 1.—

Alcohol	58.2	per cent
Gum shellac "A"	19.7	"
Pine tar oil	11.4	"
Turpentine	10.7	"

Paint No. 2.—

Same as No. 1, except 1 per cent beeswax added in place of 1 per cent of the shellac "A".

Paint No. 3.—

Same as No. 1, except that half of the shellac is replaced by yacca gum.

Paint No. 4.—

For Anti-corrosive:

Linseed oil	12.1	per cent
Volatile materials	72.3	"
Gums and resinates	15.6	"

Another way of stating its composition:

Rubbing varnish	20	per cent
Grinding japan	33	"
Mineral spirits	6.8	"
Benzol	9.2	"
Pale gloss oil	9.8	"
Light naphtha	21.2	"

For Anti-fouling:

Crude mineral oil	5.85	per cent
Pale gloss oil	81.41	"
Solvent naphtha	6.46	"
Grinding japan	6.28	"

Paint No. 5.—

Rosin	43	per cent
Ozekerite	2	"
Cumar rubber	5	"
Petroleum spirits	50	"

The cumar rubber is made from 1 part rubber, 2 parts cumar gum, 2 parts petroleum spirits.

Paint No. 6.—

For Anti-corrosive:

Ester gum	45	per cent
Spar varnish	4.6	"
Plasticized benzol	21.5	"
Benzol	28.9	"

Plasticized benzol is benzol containing 5 per cent of crude rubber.

For Anti-fouling:

Rosin	39.4	per cent
Spar varnish	4.6	"
Mineral spirits	56	"

Paint No. 7.—

For Anti-corrosive:

Rosin	12.90	per cent
Coal-tar naphtha	37.05	"
Coal tar	7.05	"
Above cumar rubber solution	6.00	"
Zinc resinate	32	"
Petroleum spirits	5	"

For Anti-fouling:

Same as Paint No. 6

Paint No. 8.—

Rosin	48	per cent
Pine tar oil	6.4	"
Coal-tar naphtha	45.6	"

Paint No. 9.—

Yacca gum	24.7	per cent
Alcohol	63.3	"
Pine tar oil	6.2	"
Turpentine	5.8	"

Paint No. 12.—

Perfection lac in place of shellac in No. 1

Paint No. 13.—

Synth-lac in place of shellac in No. 1

Paint No. 15.—

German shellac, Grade RB-1, in place of shellac in No. 1

Paint No. 16.—

Syntellac "R" in place of shellac in No. 1 paint

Paint No. 17.—

Syntellac "L" in place of shellac in No. 1 paint

Paint No. 18.—

Rosin	40	per cent
Above cumar rubber solution	20	"
Pine oil	4	"
Benzine	28	"
Mineral spirits	8	"

Paint No. 21.—

For Anti-corrosive:

Same as paint No. 7

For Anti-fouling:

Coal tar	10.1	per cent
Gasoline	13.45	"
Mineral spirits	2.5	"
Plasticized rosin	25.2	"
Zinc oxide	15.1	"
Indian red	10.1	"
Copper oxide, electrolytic	13.45	"
Mercuric oxide, yellow	10.1	"

In preparing the panels, which had been cleaned carefully by pickling so as to be rust and acid free, one coat of the anti-corrosive was first applied and the panels were permitted to dry one day. Then one coat of the anti-fouling was applied, and after a day, the panels were submerged approximately three feet below low tide.

At the inspection at Raritan Bay, there were present, Messrs. T. R. Collins, A. L. M. Holzapfel, A. C. Holzapfel, A. M. Muckenfuss, A. C. Reed, W. C. Weber and G. D. White. At the inspection at Hampton Roads, there were present, Messrs. N. E. Adamson, A. L. M. Holzapfel, A. M. Muckenfuss and G. D. White.

REPORT ON INSPECTIONS OF TEST PANELS

Best grade, 10; Poorest grade, 0.

Raritan Bay (Submerged July 25; Inspected October 28, 1925).—

Paint	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 12
Corrosion	7	6	7	7	7	7	7	7	8	3
Fouling	2	5	0	9	3	4	4	4	7	4

Paint	No. 13	No. 15	No. 16	No. 17	No. 18	No. 21
Corrosion	6	8	6	8	7	5
Fouling	8	2	7	5	5	8

Hampton Roads (Submerged August 13; Inspected October 30, 1925).—

Paint	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 13
Corrosion	6	6	5	8.5	3	5	2	5	8.5	7.5
Fouling	8.5	7.5	6	9	4	6.5	5	7	7	9

Paint	No. 15	No. 16	No. 17	No. 18
Corrosion	5	7	8	4
Fouling	9	8.5	9	7.5

Missing numbers were either not painted or the panels fell off before inspection. The total amount of fouling was much greater at Raritan Bay than at Hampton Roads.

Photographs are on file of the appearance of the panels at the time of inspection. One definite result is confirmed in this year's report, namely, that the differences in fouling and corrosion are as appreciable in under-water paints by varying the vehicle as they are by varying the pigment. This is analogous to the well-known facts with reference to above-water paints.

Respectfully submitted on behalf of the sub-committee,

A. M. MUCKENFUSS,
Chairman.

REPORT OF COMMITTEE D-2
ON
PETROLEUM PRODUCTS AND LUBRICANTS

Committee D-2 has held three regular meetings during the past year (in June at Atlantic City, in October at New York and in March at Pittsburgh), and also an informal meeting at Fort Worth, Texas, at the time of the Annual Meeting of the American Petroleum Institute, which afforded an additional opportunity for discussion of the work of various sub-committees.

Four new sub-committees have been formed during the year, as follows:

XXIV. Calorific Value,
XXV. Crankcase Dilution,
XXVI. Petroleum Wood Preservatives, and a
Special Sub-Committee on Application of Tests,

the last named sub-committee being composed of the Chairmen of all the sub-committees.

Committee D-2 now has a voting membership of 37 producers and 39 non-producers and is pleased to report the continued growth in the use of the various standards and tentative standards prepared.

NEW TENTATIVE STANDARDS

The committee is presenting for approval for publication as tentative standards, the following proposed methods of test:

1. *Method of Test for Penetration of Cup and Railroad Greases*, submitted by Sub-Committee IV on Grease and appended hereto.[1]

2. *Method of Test for Color Determination of Petrolatum by Means of the Union Colorimeter*, submitted by Sub-Committee VI on Color (I. K. Giles, Chairman) and appended hereto.[2] This method is an adaptation of the present A.S.T.M. Method for the Determination of Color of Lubricating Oils. The sub-committee is also working on a method of determining the color of wax but is not yet prepared to recommend a method for publication.

3. *Method of Test for Neutralization Number of Petroleum Products and Lubricants*, submitted by Sub-Committee XIII on Neutralization

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[1] See p. 701.—Ed. [2] See p. 713.—Ed.

Number and Saponification and appended hereto.[1] (This is a substitute for the Tentative Method of Test for Free Acid in Lubricants (D 188 - 24 T).)

4. *Method of Test for Burning Quality of Long-Time Burning Oil for Railway Use*, as submitted by Sub-Committee XIX on Illuminating Oil (J. B. Rather, chairman) and appended hereto.[2] The difficulties in the way of the development of a method satisfactory to every one were discussed in the 1924 report of the committee, and are too well known to require further comment. Nevertheless, the sub-committee felt that the only way to make progress would be to prepare a method which, upon publication, would serve as the basis for criticism. The principle of the method is fixed oil consumption and not fixed burning time. Under the conditions specified the burning time will vary from 120 to 144 hours. The reason for this lies in the fact that with any specified nominal flame height, the oil consumption for any given period will vary with very small differences in flame setting and other burning conditions.

The point has been raised in connection with the wick specified in the test, that a center-core wick might produce different results. Through the courtesy of C. P. Van Gundy, the sub-committee was supplied with a number of these wicks, but was unable to discover any differences in results on the oils available for test.

5. *Method of Test for Distillation of Natural Gas Gasoline*, submitted by Sub-Committee XXII on Natural Gas Gasoline (Lloyd F. Bayer, chairman) and appended hereto.[3] The method follows where possible the wording of the present Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 - 24 T).

RECOMMENDED REVISIONS OF TENTATIVE STANDARDS

The committee recommends certain changes in the following tentative methods of test, and upon approval of these revisions, that the methods be continued as tentative.

No. 1. *Tentative Methods of Test for Viscosity of Petroleum Products and Lubricants* (D 88 - 24 T).[4] The revisions recommended, as submitted by Sub-Committee V on Viscosity (Winslow H. Herschel, chairman), are as follows:

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[1] See p. 722.—ED. [2] See p. 718.—ED. [3] See p. 693.—ED.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 883 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 277.

Section 1.—Add to the table of dimensions:

> "Diameter of container between bottom of cylindrical part of container and top of outlet tube . . . 0.9 cm., minimum."

This limitation is to prevent the diameter being made so small as to restrict the flow.

Change footnote (*b*), Table I, from its present form: namely,

> "The section of overflow rim must show a straight line from the outside of rim at top to the bottom of gallery."

to read as follows:

> "The section of overflow rim shall be bounded by straight lines, except that a fillet is permissible at the junction with the bottom of the gallery."

The object of the fillet is to facilitate cleaning.

Section 2 (c).—In the thermometer specifications, under "stem" insert the following:

> "The stem shall preferably be made with an enlargement not less than 4 nor more than 7 mm. (0.16 to 0.28 in.) in length, having a diameter 2 to 3 mm. (0.08 to 0.12 in.) greater than that of the stem, the bottom of the enlargement being 114 mm. (4.5 in.) above the bottom of the bulb."

This enlargement is to facilitate the attachment of a guard to limit the motion of the thermometer, and it is believed that it will be of very material assistance in preventing damage both to the outlet tube of the viscosimeter and to the thermometer itself.

Section 4.—Change the first paragraph from its present form: namely,

> "Throughout the test the bath temperature shall not vary more than ±0.1° F. (0.06° C.) from the predetermined temperature which will maintain thermal equilibrium until the oil tube thermometer is withdrawn. Water shall be used as the bath liquid at all temperatures.[1]"

to read as follows:

> "In tests at 100 and 130° F. (37.8 and 54.4° C.), the bath temperature, throughout the test, shall not vary more than ±0.1° F. (0.06° C.) from the predetermined temperature which will maintain thermal equilibrium until the oil tube thermometer is withdrawn. In tests at 210° F. (98.9° C.) a variation of ±0.2° F. (0.12° C.) is permitted. Water shall be used as the bath liquid at 100 and 130° F. (37.8 and 54.4° C.).[1]"

In the second paragraph, change the third sentence from its present form: namely,

> "The heating and stirring of the water bath for the determination of viscosity at 210° F. (98.9° C.) shall be accomplished by the direct injection of steam near the bottom of the bath."

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[1] Retain the present footnote without change.

to read as follows:

> "In tests at 210° F. (98.9° C.) the water shall be vigorously stirred, and may be heated by the direct injection of steam near the bottom of the bath. A bath of other suitable liquid may be used provided it is properly heated and stirred.[a]"

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[a] For example, the bath liquid may be water, to which glycerin or salt has been added.

The last two changes are recommended to obviate difficulties experienced under certain barometric conditions, with the specified method of temperature control at 210° F.

A possible restriction of the amount of metal in the rim of the overflow cup projecting above the cover of the bath, in order to prevent excessive radiation, is under consideration.

No. 2. *Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine and Similar Petroleum Products (D 86 - 24 T).*[1]—The revisions recommended, as submitted by Sub-Committee VIII on Distillation (E. W. Dean, chairman) are as follows:

Section 7.—Change the next to last sentence from its present form: namely,

> "The distance from the 100-cc. mark to the rim shall be not less than 1¼ in. (3.175 cm.) nor more than 1¾ in. (4.445 cm.)."

to read as follows:

> "The overall height of the graduate shall be not less than 9¾ in. (24.8 cm.), nor more than 10¼ in. (26.0 cm.)."

Section 9.—Change the last sentence of the first paragraph and the whole of the second paragraph, from their present form: namely,

> "The reading of the distillation thermometer shall be recorded when the level of the distillate reaches each 10-cc. mark on the graduate.
>
> "After the 90-per-cent point has been recorded, the heat may be increased because of the presence of the heavy ends which have high boiling points. However, no further increase of heat should be applied after this adjustment. The 4 to 5-cc. rate can rarely be maintained from the 90-per-cent point to the end of the distillation, but in no case should the period between the 90-per-cent and the end point be more than five minutes."

to read as follows:

> "The volume of distillate collected in the cylinder shall be observed and recorded, to the nearest 0.5 cc., when the mercury of the thermometer reaches each point that is an even multiple of 25° C. or the Fahrenheit equivalent of this point (50° C., 75° C., 100° C., 125° C., etc., or 122° F., 167° F., 212° F., 257° F., etc.).
>
> "When the liquid residue in the distillation flask is approximately 5 cc. the heat may be increased because of the presence of heavy ends which have

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 864 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 258.

relatively high boiling points. However, no further increase of heat shall be applied after this adjustment. The 4 to 5-cc. rate can rarely be maintained from this point to the end of the distillation, but in no case shall the period between the point when approximately 5 cc. of liquid remains in the flask and the end point be more than 5 minutes."

Section 11.—Add a new Section 11 to read as follows:

CORRECTION FOR BAROMETRIC PRESSURE

"11. The actual barometric pressure shall be ascertained and recorded, but no correction shall be made except in case of dispute. In such cases the temperature points shall be corrected to 760 mm. (29.92 in.), by the use of the Sydney Young equation, as follows:

For centigrade readings:

$$C_c = 0.00012\ (760-P)\ (273 + t_c)$$

For Fahrenheit readings:

$$C_f = 0.00012\ (760-P)\ (460 + t_f)$$

in which C_c and C_f are, respectively, corrections to be added to the observed temperature t_c or t_f, and P is the actual barometric pressure in millimeters of mercury.

"The following table is a convenient approximation of the corrections as calculated by the above equation.

Temperature Range, deg. Cent.	Temperature Range, deg. Fahr.	Correction[1] per 10-mm. Difference in Pressure, deg. Cent.	Correction[1] per 10-mm. Difference in Pressure, deg. Fahr.
10 - 30	50 - 86	0.35	0.63
30 - 50	86 - 122	0.38	0.68
50 - 70	122 - 158	0.40	0.72
70 - 90	158 - 194	0.42	0.76
90 - 110	194 - 230	0.45	0.81
110 - 130	230 - 266	0.47	0.85
130 - 150	266 - 302	0.50	0.89
150 - 170	302 - 338	0.52	0.94
170 - 190	338 - 374	0.54	0.98
190 - 210	374 - 410	0.57	1.02
210 - 230	410 - 446	0.59	1.06
230 - 250	446 - 482	0.62	1.11
250 - 270	482 - 518	0.64	1.15
270 - 290	518 - 554	0.66	1.19
290 - 310	554 - 590	0.69	1.24
310 - 330	590 - 626	0.71	1.28
330 - 350	626 - 662	0.74	1.32
350 - 370	662 - 698	0.76	1.37
370 - 390	698 - 734	0.78	1.41
390 - 410	734 - 770	0.81	1.45

No. 3. *Tentative Method of Test for Cloud and Pour Points of Petroleum Products (D 97 - 23 T).*[2]—The following revisions are presented on the recommendation of Sub-Committee XVI on Cloud and Pour Test (R. R. Matthews, chairman):

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[1] To be added in case barometric pressure is below 760 mm.; to be subtracted in case barometric pressure is above 760 mm.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 698 (1923); also 1924 Book of A.S.T.M. Tentative Standards, p. 319.

Section 9.—After the present second sentence, insert a new sentence to read as follows:

"For determination of very low pour points a smaller insulated cooling bath may be used and the test jar placed directly in it."

Change the temperatures in the note on freezing mixtures to read as follows:

"For temperatures down to 50° F., ice and water.

"For temperatures down to 10° F., crushed ice and sodium chloride.

"For temperatures down to −15° F., crushed ice and calcium chloride.

"For temperatures down to −70° F., solid carbon dioxide and acetone or gasoline."

Section 10.—Change the figures in the first sentence of the second paragraph, so as to make the sentence read as follows:

"The clear oil shall be poured into the test jar, *a*, to a height of not less than 2 nor more than 2¼ in."

Section 11.—Change the first paragraph to read as follows by the addition of the italicized words and the omission of those in brackets:

"[The oil to be tested shall be brought to a temperature of 90° F., or to a temperature 15° F. higher than its pour point if this pour point is above 75° F.] The oil shall [then] be poured into the test jar, *a*, to a height of not less than 2 nor more than 2¼ in.[1] *When necessary, the oil shall be heated in a water bath just sufficiently for pouring into the test jar.* [The jar may be marked to indicate the proper level.]"

Eliminate the footnote printed in connection with the above paragraph, reading as follows:

"[1] Test jar and contents shall be immediately placed in the previously assembled apparatus."

After the present second paragraph of Section 11 insert a new paragraph to read as follows:

"Heat without stirring to a temperature of 115° F. in a bath maintained at not higher than 118° F. The oil shall then be cooled to 90° F. in air or in a water bath approximately 77° F. in temperature. Oils with which the low cloud and pour test thermometer can be used from the beginning of the test shall be cooled to 60° F. in any convenient manner before placing that thermometer in position."

Change the first sentence of the sixth paragraph to read as follows, by the addition of the italicized words and figures:

"*Beginning at a temperature 20° F. before the expected pour point,* at each test thermometer reading which is a multiple of 5° F., the test jar shall be removed from the jacket carefully and shall be tilted just sufficiently to ascertain whether there is a movement of the oil in the test jar."

After the present first sentence in the seventh paragraph insert a new sentence to read as follows:

"Certain lubricating oils tend to move as a whole and should be very closely observed."

Add a new Section 12 to read as follows:

SPECIAL PROCEDURE FOR BLACK OILS AND CYLINDER STOCKS

"12. (*a*) In those cases where it is known that a sample has been subjected to some temperature higher than 115° F. during the preceding 24 hours, or where the history of the sample in this respect is not known, the sample shall be held in the laboratory twenty-four hours before testing, unless three consecutive tests of the same sample in the same test jar give check results. For these particular oils this shall be called the maximum pour point.

"(*b*) The minimum pour point shall be determined by heating a sample with stirring to 220° F. The oil shall then be poured in the test jar, cooled to 90° F. as before, and the pour point determined as described in Section 11.

"(*c*) The pour point of such oils shall be reported as lying between the maximum and minimum pour points (*e. g.*, Pour Point 35° F./50° F.)."

Add the following requirements for a special low-cloud and pour test thermometer:

"These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −60 to +20° C. or −70 to +70° F.

TYPE: Etched stem, glass.

LIQUID: Toluene or other suitable liquid colored red with a permanent dye.

RANGE AND SUB-DIVISION: −60 to +20° C. in 1° C. or −70 to +70° F. in 2° F.

TOTAL LENGTH: 227 to 231 mm. (8.92 to 9.08 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter 7.0 to 8.0 mm. (0.28 to 0.31 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 8.0 to 9.5 mm. (0.32 to 0.37 in.).
Diameter, 5.0 to 6.5 mm. (0.22 to 0.26 in.).

DISTANCE TO −57° C. or −70° F. LINE FROM BOTTOM OF BULB: 120 to 130 mm. (4.73 to 5.12 in.).

DISTANCE TO +20° C. or +68° F. LINE FROM TOP OF THERMOMETER: 35 to 45 mm. (1.38 to 1.77 in.).

EXPANSION CHAMBER: To permit heating to +60° C. or +140° F.

FILLING ABOVE LIQUID: Gas under pressure.

TOP FINISH: Plain.

GRADUATION: All lines, figures and letters clear cut and distinct, lines at multiples of 5° C. or 10° F. to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

IMMERSION: 108 mm. (4¼ in.). The words "108-mm. immersion" on Centigrade thermometers, or "4¼-in. immersion" on Fahrenheit thermometers and a line around the stem 108 mm. (4.25 in.) above the bottom of the bulb, shall be etched on the thermometer.

SPECIAL MARKING: "A.S.T.M. Low Cloud and Pour" a serial number and the manufacturer's name or trade mark shall be etched on the thermometer.

SCALE ERROR: The error at any point of the scale, when the thermometer is standardized as provided below, shall not exceed 1° C. or 2° F.

STANDARDIZATION: The thermometer shall be standardized at the ice point and at intervals of approximately 20° C. or 35° F. for 108-mm. or 4¼-in. immersion, and for an average temperature of 21° C. or 70° F. for the emergent liquid column.

CASE. The thermometer shall be supplied in a suitable case on which shall appear the marking "A.S.T.M. Low Cloud and Pour," −60 to +20° C. or −70 to +70° F. according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications, the following definitions apply:

The total length is the overall length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

No. 4. *Tentative Methods of Testing Gas Oils (D 158 - 23 T).*[1]—The revisions recommended, as submitted by Sub-Committee XVIII on Gas Oil (C. A. Lunn, chairman) are as follows:

Section 8 (b).—Change the next to the last sentence from its present form: namely,

"The distance from the 100-cc. mark to the rim shall be not less than 1¼ in. (3.18 cm.) nor more than 1¾ in. (4.45 cm.)."

to read as follows:

"The overall height of the graduate shall be not less than 9¾ in. (24.8 cm.) nor more than 10¼ in. (26.0 cm.)."

Section 9 (c).—Omit the note relating to methods of dehydrating and measuring the sample for distillation.

Section 10.—Change to read as given below. The revisions consist of changes in the order of the italicized sentences to obtain a more logical arrangement, elimination of the entire paragraph covering "cracking"; the substitution of a footnote to deal with "cracking"; and the addition of a footnote to deal with corrections for barometric pressure.

"*Distillation.*—When everything is in readiness, water shall be circulated through the condenser bath at such a rate that the water overflowing the condenser shall be at a temperature of 90 to 100° F. (32.2 to 37.8° C.). Heat shall be applied to the contents of the flask at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 10 nor more than 15 minutes. When the first drop falls from the end of the condenser, the reading of the distillation thermometer shall be recorded as the initial boiling point. The receiving cylinder shall then be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the distillation will proceed at a uniform rate of not less than 8 cc.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 703 (1923); also 1924 Book of A.S.T.M. Tentative Standards, p. 324.

nor more than 10 cc. per minute. A fraction shall be separated at every temperature point above the initial boiling point that is a multiple of 50° F.[a] When the temperature of the distilling vapors reaches 500° F. (260° C.), the temperature of the water in the condenser bath shall be adjusted to approximately 140° F. and the bath maintained between 140 and 160° F. (60 to 71.1° C.) for the remainder of the test. The distillation shall be continued until a vapor temperature of 700° F. (371.1° C.) is reached or until the maximum temperature point is reached. *The maximum temperature point may result from complete distillation of the oil below 700° F. (371.1° C.), or from cracking.*[b]

"In case the oil distills completely below 700° F. (371.1° C.), the final adjustment of heat shall be made when a quantity of distillate amounting to 90 per cent of the sample has been collected. The heating shall be continued until the mercury reaches a maximum height and starts to fall consistently; but in no case should the period between the 90-per-cent point and the maximum temperature be more than 10 minutes. The highest temperature observed on the distillation thermometer shall be recorded as the maximum temperature.

"*Sufficient time shall be allowed after distillation is discontinued for the condenser to drain into the final fraction.* Each fraction shall be brought to room temperature, the volume read and recorded, and the fraction set aside for the determination of the gravity. The percentage of distillate in each fraction shall be calculated and recorded. The difference between 100 and the sum of the distillates in per cent shall be calculated and recorded as residue and loss.

"The gravity of each fraction and of the residue shall be determined at or corrected to 60° F. (15.6° C.). The gravity of each fraction shall be recorded as specific gravity or as gravity, degrees A. P. I."

The paragraph to be eliminated is the following:

"Cracking will be evidenced by an increase in distilling rate with a thermometer reading that may advance very slowly, remain stationary, or recede, and an effort to adjust the distilling rate will usually result in a decided drop in the temperature reading. When this condition is observed, distillation shall be discontinued and the preceding fraction considered as the final fraction."

Section 14.—Change to read as follows. This revision eliminates all mention of Furol Viscosity and the determination of Universal Viscosity at 122° F.:

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[a] When it is agreed to make allowance for the effect of barometric pressure, the observed temperature points at which fractions are to be separated in order to conform to multiples of 50° F. at 760 mm. shall be calculated by the Sydney Young equation, so expressed as to simplify the application:

$C_f = 0.00012\ P(460 + t_f)$,

in which C_f is the correction to be added to the observed temperature when the barometer is above 760 mm. and to be subtracted from the observed temperature when the barometer is below 760 mm.

P is the difference in millimeters of mercury (at 32° F.) between 760 mm. and the observed barometric pressure, corrected for instrumental errors.

t_f is the temperature point to be corrected.

[b] Cracking will be evidenced by an increase in distilling rate with a thermometer reading that may advance very slowly, remain stationary, or recede, and an effort to adjust the distilling rate will usually result in a decided drop in the temperature reading. When this condition is observed, it is advisable to discontinue the distillation.

"The viscosity shall be determined on the Saybolt Universal Viscosimeter at 100° F. in accordance with the Tentative Method of Test for Viscosity of Petroleum Products and Lubricants (Serial Designation: D 88 - 24 T) of the American Society for Testing Materials."

Since the adoption of the present tentative methods, Sub-Committee XX on Nomenclature has defined "gas oil" as "A liquid petroleum distillate having viscosity intermediate between that of kerosine and lubricating oil." The proposed changes in the tentative methods eliminate those provisions which are not believed to be necessary for the testing of gas oils as defined above.

WITHDRAWAL OF TENTATIVE STANDARDS

The committee recommends the withdrawal of Tentative Method of Test for Free Acid in Lubricants (D 188 - 24 T), in accordance with the recommendation of Sub-Committee XIII on Neutralization Number and Saponification contingent upon the approval of the proposed tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants.

NEW STANDARD

An Abridged Volume Correction Table for Petroleum Oils, submitted by Sub-Committee XV on Sampling and Gaging, and appended hereto,[1] is recommended for adoption as standard without its first being published as tentative. The committee accordingly asks for the necessary nine-tenths vote on this adoption.

Appended hereto are reports of the following sub-committees:

- Sub-Committee III on Paraffin Wax.
- Sub-Committee IV on Grease;
- Sub-Committee VII on Sulfur Determination and Differentiation;
- Sub-Committee XI on Emulsification;
- Sub-Committee XIII on Neutralization Number and Saponification;
- Sub-Committee XV on Sampling and Gaging;
- Sub Committee XX on Nomenclature;
- Sub-Committee XXI on Crude Petroleum;
- Sub-Committee XXIV on Calorific Value.

Sub-Committee X on Oxidation at High Temperatures under the chairmanship of H. G. Smith has directed its attention for the most part to the investigation of the oxidation test of lubricating oils developed by T. S. Sligh, Jr., of the Bureau of Standards, and described in a paper presented by him at the 1924 annual meeting.[1]

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[1] See A.S.T.M. Standards Adopted in 1925.—ED.

The results so far indicate reasonable reproducibility, which although not perfectly satisfactory, is appreciably better than secured by some other methods now in use. Further work will be carried on in an endeavor to very carefully control various factors which may introduce variation, and the sub-committee is also cooperating with Sub-Committee IX on Precipitation in the preparation of a suitable naphtha for use in the oxidation test.

In addition, work is planned on a test proposed by Mr. Smith somewhat similar to the Sludging Value Test for Transformer and Switch Oils now the standard of the Institution of Petroleum Technologists and of the British Engineering Standards Association.

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 76 members; 43 ballots have been returned, and 33 members have refrained from voting. The result of the letter ballot is as follows:

Item	Affirmative	Negative	Not Voting
I. PROPOSED NEW TENTATIVE STANDARDS			
Method of Test for Penetration of Cup and Railroad Greases	36	3	4
Method of Test for Color Determination of Petrolatum by Means of the Union Colorimeter	35	0	8
Method of Test for Neutralization Number of Petroleum Products and Lubricants	37	1	5
Method of Test for Burning Quality of Long-Time Burning Oil for Railway Use	37	0	6
Method of Test for Distillation of Natural Gas Gasoline	34	0	9
II. PROPOSED REVISION OF EXISTING TENTATIVE STANDARDS			
Method of Test for Viscosity of Petroleum Products and Lubricants (D 88 - 24 T)	41	1	1
Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 - 24 T)	37	2	4
Method of Test for Cloud and Pour Points of Petroleum Products (D 97 - 23 T)	38	1	4
Methods of Testing Gas Oils (D 158 - 23 T)	33	1	9
III. WITHDRAWAL OF EXISTING TENTATIVE STANDARDS			
Method of Test for Free Acid in Lubricants (D 188 - 24 T)	37	1	5
IV. PROPOSED NEW STANDARD			
Abridged Volume Correction Table for Petroleum Oils	38	1	4

This report has been submitted to letter ballot of the committee, which consists of 76 voting members, of whom 43 have voted affirmatively, none negatively, and 33 have refrained from voting.

Respectfully submitted on behalf of the committee,

C. P. VAN GUNDY,
Chairman.

VAN. H. MANNING,
Secretary.

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[1] T. S. Sligh, Jr., "An Oxidation Method for Measuring the Stability of Mineral Oils," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 964 (1924).

EDITORIAL NOTE

The proposed Abridged Volume Correction Table for Petroleum Oils submitted for the first time in 1925, as mentioned in the report, was approved at the annual meeting by a nine-tenths vote and subsequently adopted by letter ballot of the Society on August 28, 1925, and appears in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

The proposed Method of Test for Penetration of Cup and Railroad Greases; for Color of Petrolatum by Means of the Union Colorimeter; for Neutralization Number of Petroleum Products and Lubricants; for Burning Quality of Long-Time Burning Oil for Railway Use; and for Distillation of Natural Gas Gasoline were accepted for publication as tentative and appear on pages 713–724 and 693–706.

The proposed revisions of the Tentative Method of Test for Viscosity of Petroleum Products and Lubricants; for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products; and for Cloud and Pour Points of Petroleum Products; and of the Tentative Methods of Testing Gas Oils were accepted. The tentative methods as thus revised appear on pages 707, 685, 725 and 732, respectively.

The withdrawal of the Tentative Method of Test for Free Acid in Lubricants was approved.

REPORT OF SUB-COMMITTEE III ON PARAFFIN WAX

As stated in the sub-committee's report for 1924, the sub-committee has abandoned its attempt to standardize the old Press Method for determination of oil and moisture in paraffin wax, and has, during the past year, confined its experiments to careful investigation of the Solvent Index Method originated by Messrs. Wilson and Wilkin of the Standard Oil Co. of Indiana.

A series of tests have been made on sets of wax samples prepared from fully refined high melting point wax with varying percentages of slack wax added to give increased oil content. As will be noted from Table I, seven of the eight laboratories reporting were in close agreement. One laboratory, through some error, reported oil contents very much above the average.

The sub-committee feels that the Solvent Index Method should be given as wide publicity as possible and that frank criticism should be invited from the membership of the Society. This is particularly desirable since the figures for oil content in a given wax by the Index of Refraction Method are much higher than those obtained by the Press Method and the eventual adoption of the Index of Refraction Method by the Society and by the industry at large, will necessitate a readjustment of the present wax standards for oil contents.

The sub-committee is accordingly presenting for information and criticisms the proposed method for the determination of oil in wax, appended hereto, anticipating that with the suggestions and criticisms received during the coming year, it will be in a position to recommend a method based on this general principle for publication as a tentative standard in 1926.

Respectfully submitted on behalf of the sub-committee,

E. B. Phillips,
Chairman.

TABLE I.—COMPILATION OF RESULTS OF TESTS OF WAX OF VARYING OIL CONTENT.

Laboratory	Weight of Sample, g.	Solvent and Residue, mg.	Refractive Index at 77° F.	Oil in Residue and Solvent, per cent	Oil in Wax, per cent	Average
SAMPLE A						
No. 1	5.0	308	1.4492	6.0	0.37	
	5.0	430	1.4485	4.2	0.36	
	5.0	711	1.4483	3.6	0.51	0.41
No. 2	4.9691	374.3	1.4497	7.3	0.55	
	5.0133	349.5	1.4497	7.3	0.51	
	5.0323	326.8	1.4502	8.5	0.55	
	5.3302	294.5	1.4509	10.2	0.56	
	5.0252	376.7	1.4498	7.6	0.57	0.55
No. 3	5.0	466	1.4495	6.8	0.63	
	5.0	394	1.4500	8.0	0.63	
	5.0	408	1.4508	9.3	0.76	0.67
No. 4	5.0	612	1.4479	2.5	0.31	
	5.0	242	1.4500	8.0	0.38	0.345
No. 5	5.320	547	1.4481	3.1	0.32	
	5.920	560	1.4488	5.0	0.47	
	5.370	598	1.4488	5.0	0.56	0.45
No. 6	5.0	257	1.4488	5.0	0.26	
	5.0	258	1.4494	6.6	0.34	0.40
No. 7	5.0	259	1.4498	7.5	0.39	
	5.0	247	1.4491	5.7	0.30	0.345
SAMPLE B						
No. 1	5.0	354	1.4522	13.4	0.96	
	5.0	1707	1.4481	13.1	1.06	1.005
No. 2	5.0112	367.1	1.4517	12.2	0.89	
	5.0075	443.8	1.4532	15.8	1.40	
	5.0620	438.5	1.4517	12.2	1.06	
	5.0974	396.4	1.4513	11.2	0.87	
	5.0026	407.7	1.4509	10.2	0.83	1.01
No. 3	5.0	479	1.4525	14.1	1.35	
	5.0	409	1.4516	11.9	0.97	
	5.0	404	1.4520	12.9	1.04	1.12
No. 4	5.0	424	1.4511	10.7	0.91	
	5.0	350	1.4522	13.4	0.94	0.925
No. 5	5.068	573	1.4502	8.5	0.96	
	4.701	570	1.4502	8.5	1.03	0.995
No. 6	5.0	310	1.4523	13.6	0.84	
	5.0	319	1.4523	13.6	0.87	0.855
No. 7	5.0	278	1.4529	15.0	0.85	
	5.0	299	1.4525	14.1	0.84	0.835

TABLE I (*Continued*).—RESULTS OF TESTS ON WAX.

Laboratory	Weight of Sample, g.	Solvent and Residue, mg.	Refractive Index at 77° F.	Oil in Residue and Solvent, per cent	Oil in Wax, per cent	Average
SAMPLE C						
No. 1	5.0	361	1.4544	18.8	1.36	
	5.0	470	1.4527	14.6	1.37	1.365
No. 2	5.1997	415.4	1.4541	18.0	1.44	
	5.1531	440.7	1.4540	17.8	1.52	
	5.0620	496.2	1.4516	11.7	1.15	
	5.0244	437.5	1.4528	14.8	1.29	
	5.0556	426.4	1.4523	13.6	1.15	1.31
No. 3	5.0	471	1.4527	14.6	1.37	
	5.0	467	1.4530	15.3	1.43	
	5.0	473	1.4527	14.6	1.38	1.39
No. 4	5.0	409	1.4538	17.3	1.42	
	5.0	399	1.4543	18.5	1.48	1.45
No. 5	5.368	592	1.4517	12.2	1.34	
	5.058	588	1.4519	12.6	1.46	1.40
No. 6	5.0	347	1.4548	20.0	1.39	
	5.0	344	1.4553	20.9	1.44	1.415
No. 7	5.0	322	1.4558	22.2	1.42	
	5.0	343	1.4548	20.0	1.37	1.395
SAMPLE D						
No. 1	5.0	571	1.4552	20.7	2.36	
	5.0	639	1.4543	18.5	2.36	2.36
No. 2	5.1203	399.9	1.4567	24.4	1.91	
	5.2753	462.7	1.4567	24.4	2.14	
	5.1850	430.3	1.4562	23.2	1.92	
	5.0086	419.5	1.4553	21.0	1.76	
	5.1275	501.6	1.4550	20.2	1.98	1.94
No. 3	5.0	526	1.4543	18.5	1.95	
	5.0	501	1.4548	19.7	1.97	
	5.0	504	1.4548	19.7	1.98	1.97
	6.8088	552	1.4548	19.7	1.59	
	6.3651	560	1.4543	18.5	1.62	1.605
	5.0	576	1.4542	18.2	2.09	
	5.0	496	1.4547	19.5	1.94	2.015
No. 4	5.0	474	1.4560	22.7	2.15	
	5.0	428	1.4570	25.1	2.15	2.15
No. 5	5.005	669	1.4538	17.3	2.31	
	5.030	661	1.4538	17.3	2.28	2.295
No. 6	5.0	363	1.4580	27.6	2.00	
	5.0	377	1.4573	25.9	1.95	1.976
No. 7	5.0	387	1.4565	23.9	1.95	
	5.0	387	1.4572	25.6	1.98	1.965

APPENDIX

PROPOSED METHOD FOR DETERMINATION OF OIL IN WAX

1. *Scope.*—This method is put forward as the best means yet devised of determining the oil content of all commercial grades of paraffin wax.

I. APPARATUS AND REAGENTS

2. (*a*) *Refractometer.*—Abbe or similar type capable of being read to the fourth decimal place for indices of refraction on small samples at constant temperature.

(*b*) *Cooling Bath.*—Cooling bath of suitable size and design capable of being maintained at 0° F. (−18° C.) or below for long periods.

(*c*) *Suction Filter.*—Suction filter of suitable design fitted with a cooling bath capable of being maintained at 0° F. (−18° C.) or below for long periods.

3. (*a*) *Primary Solvent.*—Ethylene dichloride, an 85-per-cent intermediate cut from technically pure grade of the solvent.

(*b*) *Secondary Solvent.*—A mixture of heavy and light highly refined burning oils having a refractive index of exactly 1.4470 at 77° F. (25° C.).

II. SECURING AND PREPARATION OF REFEREE SAMPLES

4. All commercial shipments of paraffin wax shall be sampled by selecting a suitable number of packages from a given lot. Each of the packages shall be sampled by passing an auger or other suitable device completely through the short dimension of the package. Borings from all the packages shall be united and pulverized by passing the entire sample through a meat chopper fitted with a fine cutting plate. The total pulverized sample shall be coned and quartered until an average sample weighing approximately 500 g. is obtained. In all cases of dispute the final sample shall be divided in equal portions, one being forwarded to the seller and the other to the laboratory representing the buyer. Due to the tendency of certain grades of paraffin wax to "sweat" with age and changes of temperature, all referee samples of wax shall be stored and shipped in glass. Tests for oil content shall be made as quickly as possible after samples have been collected.

III. PROCEDURE

5. *Preparation of Sample at Time of Test.*—If the test for oil content is made immediately after sampling, the original pulverized wax may be used, but if much time elapses between the time of sampling and time of test, the referee sample shall be melted (at a temperature not greater than 20° F. (11° C.) above its melting point), thoroughly mixed, chilled to solid form and repulverized by passing through a meat chopper.

6. *Method of Test.*—Approximately five grams of the pulverized wax shall be weighed into an Erlenmeyer flask of suitable size. About 75 cc. of the warm ethylene dichloride shall be added and the flask agitated until a clear solution is obtained. The Erlenmeyer flask shall be placed in the ice and salt bath and the solvent-wax mixture allowed to chill to about 0° F. (−18° C.). Due to the high insulating property of solid wax the flask must be frequently agitated during the cooling period to remove the wax deposit from the walls. While the solvent-wax mixture is cooling, the bath of the suction filter (Holde type) shall be prepared to produce a temperature

in the filter tube of about 0° F. (−18° C.). When the solvent-wax mixture and the filter tube have reached a temperature of about 0° F. (−18° C.), the solvent-wax mixture shall be poured into the filter tube and the flask rinsed with the ethylene dichloride reagent previously chilled to 0° F. (−18° C.). After the filtrate has drained, the remaining wax shall be washed with fresh chilled ethylene dichloride until the volume of filtrate and washings reaches about 150 cc.

The filtrate and washings shall be transferred to a beaker of suitable size and evaporated to a small residue on a steam bath. A small quantity of warm ethylene dichloride shall be added and the residue transferred to a tared weighing bottle. The beaker shall be rinsed thoroughly with warm ethylene dichloride or petroleum ether. The weighing bottle shall be placed on a steam bath and all solvents evaporated off. It shall be cooled and enough of the heavy and light burning oil mixture (refractive index 1.4470) added to liquefy the residue. Usually about 0.2 cc. is required. The weight of the residue + solvent shall be determined and its refractive index observed with an Abbe or similar instrument.

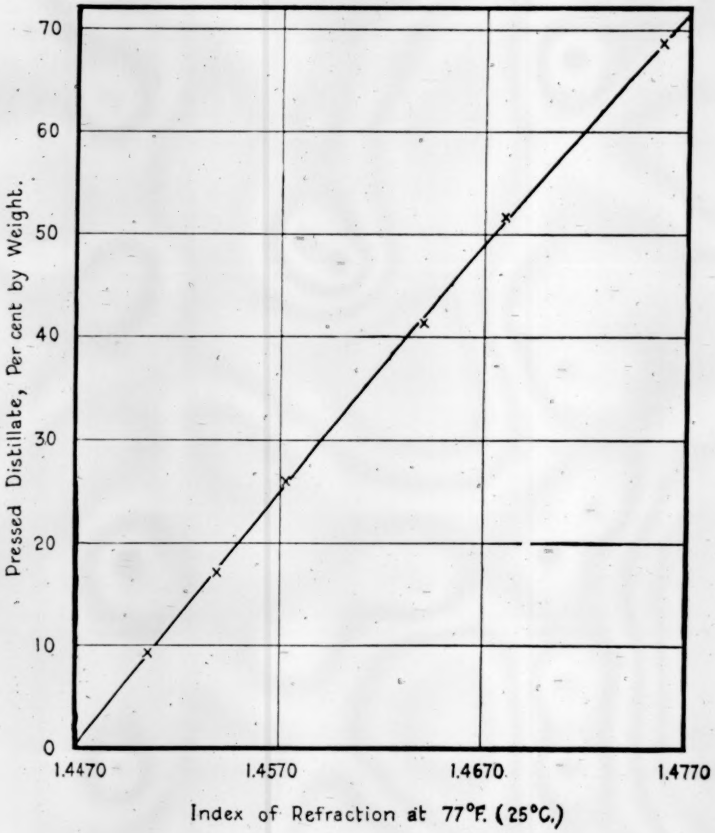

FIG. 1.

7. *Interpretation of Results.*—For a full discussion of the method see article by Wilson and Wilkins in *Industrial and Engineering Chemistry*, Vol. 16, No. 1, page 9,

January, 1924. The filtrate from the wax contains all of the oil and a little very low melting point wax. Fortunately, the index of refraction of the low melting point wax, soluble in ethylene dichloride at 0° F. (−18° C.) or below has been found to be practically constant at 1.4770 at 77° F. (25° C.) When, therefore, the residue from the extraction, free from ethylene dichloride, is dissolved in a solvent of the same index the only thing which can raise the index is the amount of oil present and since the only oil normally present is pressed distillate a curve showing the index of the solvent-wax-oil mixture as a function of the oil content can and has been prepared and is shown on a small scale in Fig. 1. For accurate work, however, a large scale curve should be constructed from the following data:

OIL IN RESIDUE, PER CENT BY WEIGHT	REFRACTIVE INDEX 77° F. (25° C.)
0	1.4470
10	1.4508
20	1.4549
30	1.4590
40	1.4631
50	1.4674
60	1.4718
70	1.4762

By use of the above curve the results obtained in the test are converted into percentage of oil by weight in the original wax sample. Use is made of the following formula:

$$\text{Percentage Oil in Wax} = \frac{\text{Weight Residue} \times \text{Percentage of Oil (from chart)}}{\text{Weight of Original Wax}}$$

8. *Reporting Tests.*—Oil in wax determined by the above method shall be reported as "Oil Content by A.S.T.M. Solvent Index Method."

REPORT OF SUB-COMMITTEE IV ON GREASE

The first problem attacked by the sub-committee on grease during the past year was the development of a method of uniformly working grease to a constant consistency. It was found to be very difficult to work the grease without incorporating considerable air into it. A worker has been designed, however, which keeps this to a minimum, and, as shown in Table I, can be relied upon to give reproducible results. This method of working and the apparatus used is described in the proposed tentative method which is being submitted.[1]

The cone for measuring the consistency specified in the proposed method is one with a double pitch instead of the 45-deg. cone previously reported on. This cone was designed to be sufficiently sensitive for hard railroad greases and at the same time satisfactory for fairly soft greases. It was constructed after considering the flaring cones submitted by the Bureau of Standards and the Standard Oil Co. of California. For testing hard greases the Bureau of Standards cone was excellent, but it submerged when an attempt was made to extend its application to No. 0 greases. The Standard Oil Co. of California cone was not sensitive enough for hard greases but could be used for soft greases. The new cone gives intermediate results and is satisfactory at both extremes.

TABLE I.—RESULTS REPORTED ON GREASE SAMPLES.

Operator	Sample "A"				Sample "B"				Sample "C"		Average of Average Deviation, per cent
	Un-worked	Average Deviation, per cent	Worked	Average Deviation, per cent	Un-worked	Average Deviation, per cent	Worked	Average Deviation, per cent	Un-worked	Average Deviation, per cent	
No. 1	256	0.9	299	0.8	100	1.9	189	1.1	19.8	3.1	1.6
No. 2	253	1.2	283	1.7	97	2.6	182	1.2	16.2	3.0	2.0
No. 3	232	0.9	280	2.9	103	1.6	181	1.2	18.6	3.5	2.0
No. 4	260	0.2	284	0.2	101	0.3	182	0.3	15.7	2.2	0.6
No. 5	256	1.3	284	0.6	102	1.4	184	1.9	18.4	3.7	1.8
No. 6	251	1.5	295	0.8	102	1.1	194	0.5	20.0	3.0	1.4
No. 7	250	0.3	282	1.6	103	1.1	191	1.3	19.0	3.7	1.6
No. 8	273	3.1	312	0.5	104	2.1	203	1.0	...	...	...
No. 9	254	0.7	299	0.5	98	9.3	203	0.9	18.3	2.4	2.8
No. 10	256	2.4	303	3.2	95	3.4	200	3.3	19.0	1.6	2.8
No. 11	232	3.9	286	2.0	98	3.8	192	1.7	20.0	3.0	2.9
No. 12	259	2.7	298	1.7	105	2.9	200	1.9	20.3	2.0	2.2
Average	253	1.6	293	1.4	101	2.6	192	1.4	18.6	2.8	2.0
Average percentage of deviation between laboratories	3.0	....	3.1	....	2.5	....	3.8	....	5.9	....	3.6

The new cone was made with a double pitch instead of a spherical flare since this type is easier to construct with accuracy and it is very simple to check it up. This cone is described in the tentative method proposed.

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[1] See p. 701.—ED.

During March, comparative tests were made by twelve members of the committee on three samples of grease furnished by the chairman's laboratory. The proposed workers and cones were also furnished. The method used was that described in the proposed tentative method. Tests were made on the greases before and after working and each member reported ten determinations on each sample. The results of these tests are recorded in Table I.

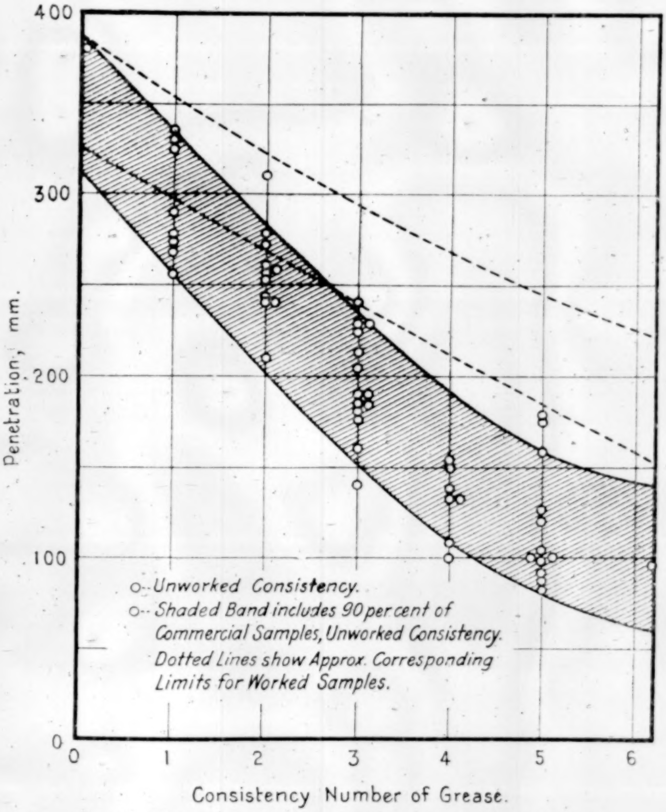

FIG. 1.—Results of Consistency Measurements of Grease from Various Manufacturers.

Considering the unavoidable variations in the consistency of different cans of grease and even of different parts of the same can, and considering further that this was the first time the grease worker and the new cone had been tried out by the committee members it is considered that the results reported show remarkably good agreement. This is especially apparent if comparison is made with some of the early attempts to standardize a consistency method on grease. Even comparing with the good results reported last year on the 45-deg. cone, the average deviation between individual observations in the given laboratory is only 2.0 per cent as compared with 2.9 per cent last year and the average mean deviation between different laboratories is 3.6 per cent as compared with 5.3 per cent previously. On the basis of these results together with the experience of the committee on similar methods during the past three years it is felt that the proposed method can be relied upon to meet the growing demand for a reliable standard consistency test for grease.

Last year's report gave penetrations with the 45-deg. cone on samples of cup grease made by ten different manufacturers. Since then some additional results have been obtained on commercial samples of grease using the new cone. The results, all converted to the double pitch cone, are shown in Fig. 1. Obviously the variations between the products of different manufacturers is quite large.

The committee recommends that the method for consistency tests on grease be made a Tentative Standard of the Society.

Respectfully submitted on behalf of the sub-committee,

ROBERT E. WILSON,
Chairman.

REPORT OF SUB-COMMITTEE VII ON SULFUR DETERMINATION AND DIFFERENTIATION

SULFUR CONTENTS OF MOTOR FUELS

Doubts have been expressed about the reliability of the Tentative Method of Test for Sulfur in Naphthas and Illuminating Oils (D 90 – 24 T) for determination of the sulfur content in motor fuels containing benzol, especially if the latter is contaminated by carbon

TABLE I.—RESULTS OF SULFUR DETERMINATIONS OF MOTOR FUELS CONTAINING BENZOL.

	Lamp Method (D 90 – 21 T)			Bomb Method		
Laboratory	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
No. 1	0.192	0.157	0.201	0.226	0.186	0.238
No. 2	0.180	0.150	0.200	0.196	0.180	0.216
	0.190	0.150	0.210	0.208	0.180	0.240
	0.200	.....	0.200	0.200	0.198	0.205
Average	0.190	0.150	0.203	0.201	0.186	0.220
No. 3	0.189	0.155	0.186	0.251	0.194	0.268
	0.195	0.149	0.180	0.211	0.212	0.252
	.....	0.152	0.171	.....	....	.....
Average	0.192	0.152	0.179	0.231	0.203	0.260
No. 4	0.15	0.042	0.300	.....	.....	.....
	0.15	0.038	0.301	.....	.....	.....
Average	0.15	0.040	0.300			
No. 5		.....	.....	0.28	Trace	0.22
No. 6	0.191	0.144	0.202	0.186	0.135	0.193
	0.197	0.145	0.201	0.180	0.131	0.201
Average	0.194	0.145	0.201	0.183	0.133	0.197
No. 7	0.190	0.119	0.220	.....	0.120	0.181
	0.194	0.120	0.225	0.180	0.124	0.191
	.....	.....	.....	0.174	0.128	0.200
Average	0.192	0.120	0.223	0.177	0.124	0.191

disulfide. Accordingly, there were sent to the members of the sub-committee, samples of this class of fuel, as follows:

1. Fuel bought in the open market.
2. Mixture of gasoline and benzol, both having very low sulfur contents, with carbon disulfide added.
3. A mixture of high-sulfur-content gasoline and benzol, free from carbon disulfide.

The above samples, which contained about 35 per cent of benzol, have been examined by Method D 90 – 24 T and by the bomb method. The results, which appear in Table I, may be summed up as follows:

The lamp method as employed by the members of the sub-committee gives, in general, results that agree well. When the sulfur is determined by the bomb method, each member apparently is able to check his own results in a satisfactory manner. When the results of the various members are compared, however, greater differences occur. It appears that the bomb method needs to be modified and standardized for the special purpose of determining sulfur in motor fuels before the influence of various sulfur compounds upon the results of the lamp test can be studied. Work along this line is suggested.

CORROSION TEST FOR TRANSFORMER OILS

The following method, which is a slight modification of the Federal Specifications Board Method 530.31, is suggested:

> Place a clean strip of mechanically polished pure sheet copper, $\frac{3}{8}$ in. wide and 3 in. long, in a clean test tube of $\frac{5}{8}$ in. outside diameter and 6 in. in length. Add 15 cc. of the oil to be tested, which will completely cover the strip. Close the tube with a vented stopper and heat in a thermostat for 3 hours at 212° F. Rinse the copper strip with sulfur-free acetone and compare it with a similar strip of freshly polished copper. Darkening or pitting indicates corrosion.
>
> Repeat the above test, heating for 5 hours at 212° F. Compare the results with those obtained when heating for 3 hours.
>
> To identify corrosion caused by sulfur, wash the corroded strip with 5 cc. of a 5-per-cent potassium cyanide solution, and test for sulfides by adding to the washings 1 cc. of a freshly made 3-per-cent solution of sodium nitroprusside. A purple color indicates the presence of sulfides. As the potassium cyanide sometimes contains sulfides, a blank test is advisable.

Two samples were forwarded to the members of the sub-committee for examination by the above method. Both were the same transformer oil; the sample No. 1 contained no free or corrosive sulfur, while sample No. 2 contained 1.5 mg. of dissolved sulfur in each 100 cc. The results reported to date are as follows:

Laboratory		Sample No. 1 Corrosion	Sample No. 1 Sulfide	Sample No. 2 Corrosion	Sample No. 2 Sulfide
No. 1	3 hrs.......	None	......	None	......
	5 hrs.......	"	......	"	......
No. 2	3 hrs.......	None	......	Present[a]	Present
	5 hrs.......	"	......	"	"
No. 3	3 hrs.......	None	......	Present	Present
	5 hrs.......	"	......	"	"
No. 6	3 hrs.......	Slight	Faint	Some	Present
	5 hrs.......	Bad	Positive	Very Bad	"
No. 7	3 hrs.......	Very Slight	None	Slight	Present
	5 hrs.......	" "	"	"	"

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[a] The longer exposure did not increase the degree of corrosion.

Respectfully submitted on behalf of the sub-committee,

E. M. JOHANSEN,
Chairman.

REPORT OF SUB-COMMITTEE XI ON EMULSIFICATION

RÉSUMÉ OF PREVIOUS WORK

The members of Sub-Committee XI have been working for several years to cover the whole range of lubricating oils, and as a result of considerable work the A.S.T.M. Method of Test for Steam Emulsion of Lubricating Oils (D 157 – 23 T) was adopted as a tentative standard. While this test, according to the general vote of the sub-committee members, appeared to serve as a satisfactory basis for the majority of lubricating oils, there was some question about its applicability to turbine oils, where a very severe emulsion test appears desirable. One of the members of the sub-committee, who has done considerable work on turbine oils, sent two samples to the members of the planning committee of this sub-committee. On the basis of preliminary results obtained by the planning committee a second set of samples was forwarded to each member of the entire sub-committee. A new set of directions based on the experience of the planning committee was circulated, covering detailed points in connection with both the Herschel and Steam Emulsion Tests. These two oils were to be run on the Funk Sludging Machine also, in order to determine what relationship exists between the classification of the oils as determined by this sludging test and as determined by the Herschel and Steam Emulsion Tests.

DATA

(*a*) *Sludging Tests.*—The two oils, marked 24-1 and 24-2, respectively, have been run in the Funk oil-sludging apparatus by both Mr. Funk and Mr. Baxter. Inasmuch as a final report has not been received from Mr. Baxter, it is impossible to include in this report the final classification which would be given these oils according to sludging tests run in separate laboratories. Mr. Funk's results, based on the percentage of "sludge and emulsion" obtained at the end of a 30-hour period, would classify oil No. 24-1 as somewhat better than oil No. 24-2.

(*b*) *Emulsion Tests.*—In the accompanying Tables I to IV is given a summary of results received to date on these two oils. The data is arranged, so far as possible, according to the date of testing, but it will be noted that the variations in results between different laboratories are probably greater than those due to changes in the oil itself. All results are reported and a general average, together with average deviation, is given for both the demulsibility and steam emulsion tests.

TABLE I.—RESULTS OF INVESTIGATION ON MODIFIED A.S.T.M. STEAM EMULSION TEST, READINGS IN SECONDS.

Laboratory	Date of Test	Oil No. 24-1		Oil No. 24-2	
		Sample Measured at Room Temp.	Sample Measured at 200° F.	Sample Measured at Room Temp.	Sample Measured at 200° F.
No. 1	Oct. 15, 1924	59 65 63 — 62	58 64 62 — 61	72 67 69 — 69	68 66 72 — 69
No. 2	Oct. 14, 1924	55 50 55 — 53	45 44 44 — 44	60 49[a] 54 67 — 58	66 65 66 .. — 66
No. 4	Oct. 15, 1924	44[a] 44 46 47[a] — 45	43[a] 43[a] 40[a] 42[a] — 42	69[a] 73[a] 73[a] 74[a] — 72	70[a] 68 69[a] 72[a] — 70
No. 5	Oct. 20, 1924	60 64 66 — 64	78 74 73 — 75	66 72 75 — 71	78 77 80 — 78
No. 6	Oct. 20, 1924	96[b,c] 70[b,c] — 83	60[b] 60[b] — 60	90[b,c] 65[b,c] — 77	65[b] 65[b] — 65
No. 7	Oct. 22, 1924	55 52 54[a] 57 56[a] — 55	66 62 62 60 60 — 62	57 64 62 66 .. — 62	62 66 66 .. .. — 65
No. 8	Oct. 24, 1924	52 50 — 51	51 52 — 51.5	57 58 — 57.5	56 55 — 55.5
No. 9	Oct. 27, 1924	60[a] 60 — 60	.. .. — ..	60[a] 60 — 60	.. .. — ..
No. 10	Oct. 31, 1924	53 54 55 — 54	55.5 55.5 .... — 55.5	54 54 54 — 54	55 55 .. — 55
No. 11	Oct. 30, 1924	59[a] 62 60 — 60	53 51 51 — 52	62 64 64 — 63	67 64 67 — 66

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[a] Total volume varies from specification. (Should be 40+3 cc.) These results are not included in the average.

[b] Steaming bath temperature too high. (Should be 67 to 78° F.) These results are not included in the average.

[c] These results are the maximum variations obtained. Check results were obtained when the oil was measured at 200° F.

[d] The results obtained by Laboratory No. 16 vary so much more from the average than do those of any of the other laboratories that an error in procedure is indicated, or that apparatus which does not meet specifications has been used, so the results are not included in the average.

TABLE I.—RESULTS OF INVESTIGATION ON MODIFIED A.S.T.M. STEAM EMULSION TEST, READINGS IN SECONDS (*Continued*).

Laboratory	Date of Test	Oil No. 24-1		Oil No. 24-2	
		Sample Measured at Room Temp.	Sample Measured at 200° F.	Sample Measured at Room Temp.	Sample Measured at 200° F.
No. 12	Nov. 11, 1924	73 69 70 — 71	60 57 55 — 57	86 92 85 — 88	60 70 67 — 66
No. 13	Nov. 14, 1924	50 58 57 — 55	55 60 62 — 59	70 64 68 — 67	69 69 68 — 69
No. 14	Nov. 22, 1924	52[b] 53[b] 54[b] — 53	54[b] 53[b] 54[b] — 54	57[b] 57[b] 57[b] — 57	57[b] 56[b] 58[b] — 57
No. 15	Oct. 24, 1924	50 54 47 51 55 .. — 51	63 62[a] 63 .. .. .. — 63	56 59 63 57 60 56 — 58	69 66 .. .. — 68
No. 16[d]	Nov. 28, 1924	35 29 39 35 — 35	20–35[a] 37 .. .. — 37	5 2 .. .. — 3.5	3 2 .. .. — 3
No. 17	Dec. 31, 1924	60 55 58 55 59 — 57	58 55 55 57 .. — 56	65 68 60 57 67 — 63	67 65 65 65 .. — 65
No. 18	Jan. 21, 1925	52 50 54 55 — 53	.. .. — ..	62 64 63 .. — 63	.. .. — ..
No. 19	Dec. 10, 1924	40 60 — 50	40 .. — 40	50 60 — 55	60 .. — 60
No. 3	Dec. 1, 1924 (Corrected Results)	64 60 — 62	68 .. — 68	70 64 — 67	78 .. — 78
Average Results		56.5	58.1	64.1	66.4
Average Deviation, seconds		5.3	6.1	6.2	4.2
Average Deviation, per cent		9.4	10.5	9.7	6.3

---

[a] Total volume varies from specification. (Should be 40 + 3 cc.). These results are not included in the average.

[b] Steaming bath temperature too high. (Should be 67 to 78° F.) These results are not included in the average.

TABLE II.—DEMULSIBILITY TESTS.

Laboratory	Date of Test	Demulsibility Values				Time for 27 cc. of Oil to Separate, min.			
		Oil No. 24-1		Oil No. 24-2		Oil No. 24-1		Oil No. 24-2	
		At 100° F.	At 130° F.	At 100° F.	At 130° F.	At 100° F.	At 130° F.	At 100° F.	At 130° F.
No. 1...	Oct. 15, 1924	810	1500	520	1560	2.0	2.0	4.0	2.0
		780	1560	520	1560	3.0	2.0	4.0	2.0
		780	1560	520	1560	3.0	2.0	4.0	2.0
		790	1540	520	1560	2.7	2.0	4.0	2.0
No. 2...	Oct. 14, 1924	400	1500	420	750	5.0	2.0	5.0	3.0
		380	1560	375	750	5.0	2.0	5.0	3.0
		460	1560	400	750	5.0	2.0	5.0	3.0
		413	1540	398	750	5.0	2.0	5.0	3.0
No. 5...	Oct. 17, 1924	1500	1620	600	1620	2.0	1.0	4.5	1.0
		1500	1620	600	1620	2.0	1.0	4.0	1.0
		1500	1620	400	1620	2.0	1.0	5.0	1.0
		1500	1620	533	1620	2.0	1.0	4.5	1.0
No. 6...	Oct. 20, 1924	1620	1620	1620[a]	1620[a]	1.0	1.0	1.0	1.0
		1620	1620	1620[a]	1620[a]	1.0	1.0	1.0	1.0
		1620	1620	1620[a]	1620[a]	1.0	1.0	1.0	1.0
		1620	1620	1620	1620	1.0	1.0	1.0	1.0
No. 7[b]..	Oct. 24, 1924	600	1560	600	1560	3.0	2.0	4.0	2.0
		780	1560	570	1560	3.0	2.0	4.0	2.0
		690	1560	585	1560	3.0	2.0	4.0	2.0
No. 8...	Oct. 24, 1924..	1560	1620	1500	1620	2.0	1.0	2.0	1.0
No. 9...	Oct. 27, 1924	500	1560	540	1500	4.0	2.0	3.0	2.0
		520	1500	500	1560	4.0	2.0	4.0	2.0
		540	1500	520	....	3.0	2.0	4.0	...
		520	....	...	....	4.0	...	...	...
		520	1520	520	1530	3.7	2.0	3.7	2.0
No. 10..	Oct. 31, 1924..	540	1620	540	1620	4.0	1.0	4.0	1.0
No. 11..	Nov. 7, 1924.	720	1620	405	1560	3.0	1.0	4.0	2.0
		720	1620	...	1560	3.0	1.0	...	2.0
		720	1620	405	1560	3.0	1.0	4.0	2.0
No. 12..	Nov. 13, 1924	900	1620	1020	1620	4.0	1.0	3.0	1.0
		960	1620	1080	1620	4.0	1.0	4.0	1.0
		930	1620	1050	1620	4.0	1.0	3.5	1.0
No. 13..	Nov. 14, 1924	570	780	360	630	4.0	3.0	5.0	3.0
		540	720	330	600	4.0	3.0	6.0	4.0
		600	750	330	600	3.0	3.0	6.0	3.0
		570	750	340	610	3.7	3.0	5.7	3.3
No. 16[c].	Nov. 28, 1924	480	810[d]	180	1620	3.5	2.0	8.0	1.0
		540	810	270	900	3.0	2.0	6.0	2.0
		500	780	270	900	3.8	2.3	6.0	2.0
		507	800	240	1140	3.4	2.1	6.7	1.7

[a] Oil globules adhering to walls of the cylinder; not included in the average.

[b] The dimensions of the paddle and cylinders used in these tests were within the limits set by the Government.

[c] Standard cylinder unobtainable. These results are not included in the average.

[d] Bubbles in oil.

TABLE II.—DEMULSIBILITY TESTS (*Continued*).

Laboratory	Date of Test	Demulsibility Values				Time for 27 cc. of Oil to Separate, min.			
		Oil No. 24-1		Oil No. 24-2		Oil No. 24-1		Oil No. 24-2	
		At 100° F.	At 130° F.	At 100° F.	At 130° F.	At 100° F.	At 130° F.	At 100° F.	At 130° F.
No. 17..	Dec. 2, 1924	405	1620	405	1560	4.0	1.0	4.0	2.0
		540	1620	405	810	3.0	1.0	4.0	2.0
		405	1080	600	1500	4.0	2.0	4.0	2.0
		660	1200	450	1440	3.0	2.0	4.0	2.0
		540	1620	600	1200	3.0	1.0	4.0	2.0
		460	1620	540	1080	4.0	1.0	4.0	2.0
		502	1460	500	1265	3.5	1.3	4.0	2.0
No. 3...	Dec. 1, 1924	540	1260	360	720	3.0	2.0	5.0	3.0
		540	1200	315	540	3.0	3.0	6.0	4.0
		600[e]	....	...	...	3.0	...	...	...
		560	1230	338	630	3.0	2.5	5.5	3.5
No. 18..	Jan. 21, 1925..	780	1560	570	1200	...	...	...	...
No. 19..	Dec. 10, 1924	660	1560	720	1320	3.0	2.0	3.0	2.0
		540	1560	840	1260	3.0	2.0	3.0	2.0
		600	1560	780	1290	3.0	2.0	3.0	2.0
Average	Results......	780	1470	560	1270	3.1	1.6	4.2	2.0

[e] This value was obtained on rerunning test No. 1 after allowing it to stand while No. 2 was being run. Not included in average.

TABLE III.—SUMMARY OF AVERAGE RESULTS OF TABLE I, READINGS IN SECONDS.

Laboratory	Date of Test	Oil No. 24-1		Oil No. 24-2	
		Sample Measured at Room Temp.	Sample Measured at 200° F.	Sample Measured at Room Temp.	Sample Measured at 200° F.
No. 1	Oct. 15, 1924	62	61	69	69
No. 2	Oct. 14, 1924	53	44	58	66
No. 4	Oct. 15, 1924	45	42	72	70
No. 5	Oct. 20, 1924	64	75	71	78
No. 6	Oct. 20, 1924	83	60	77	65
No. 7	Oct. 22, 1924	55	62	62	65
No. 8	Oct. 24, 1924	51	51.5	57.5	55.5
No. 9	Oct. 27, 1924	60	..	60	..
No. 10	Oct. 31, 1924	54	55.5	54	55
No. 11	Oct. 30, 1924	60	52	63	66
No. 12	Nov. 11, 1924	71	57	88	66
No. 13	Nov. 14, 1924	55	59	67	69
No. 14	Nov. 22, 1924	53	54	57	57
No. 15	Oct. 24, 1924	51	63	58	68
No. 16	Nov. 28, 1924	35	37	4	3
No. 17	Dec. 31, 1924	57	56	63	65
No. 3	Dec. 1, 1924 (Corrected Results)	62	68	67	78
No. 18	Jan. 21, 1925	53	..	63	..
No. 19	Dec. 10, 1924	50	40	55	60

COMMENTS

The following comments by individual members are given:

Several members in their comments on the steam emulsion test favor measuring the sample at room temperature rather than at 200° F., as the slightly larger volume when measured at room temperature insures a clearer line of demarcation when 20 cc. of oil have separated and thus makes the test more accurate. One member, however, reports poor check results when the sample is measured at room temperature. It is also believed by another member that the control of the settling bath from 200 to 203° F. is close enough and is within the accuracy of the rest of the test. One member had trouble in noting the line of demarcation.

TABLE IV.—SUMMARY OF AVERAGE RESULTS OF TABLE II.

Laboratory	Date of Test	Demulsibility Values				Time for 27 cc. of Oil to Separate, min.			
		Oil No. 24-1		Oil No. 24-2		Oil No. 24-1		Oil No. 24-2	
		At 100° F.	At 130° F.	At 100° F.	At 130° F.	At 100° F.	At 130° F.	At 100° F.	At 130° F.
No. 1	Oct. 15, 1924	790	1540	520	1540	2.7	2.0	4.0	2.0
No. 2	Oct. 14, 1924	413	1540	398	750	5.0	2.0	5.0	3.0
No. 5	Oct. 17, 1924	1500	1620	533	1620	2.0	1.0	4.5	1.0
No. 6	Oct. 20, 1924	1620	1620	1620	1620	1.0	1.0	1.0	1.0
No. 7	Oct. 24, 1924	690	1560	585	1560	3.0	2.0	4.0	2.0
No. 8	Oct. 24, 1924	1560	1620	1500	1620	2.0	1.0	2.0	1.0
No 9	Oct. 27, 1924	520	1520	520	1530	3.7	2.0	3.7	2.0
No. 10	Oct. 31, 1924	540	1620	540	1620	4.0	1.0	4.0	1.0
No. 11	Nov. 7, 1924	720	1620	405	1560	3.0	1.0	4.0	2.0
No. 12	Nov. 13, 1924	930	1620	1050	1620	4.0	1.0	3.5	1.0
No. 13	Nov. 14, 1924	570	750	340	610	3.7	3.0	5.7	3.3
No. 16	Nov. 28, 1924	507	800	240	1140	3.4	2.1	6.7	1.7
No. 17	Dec. 2, 1924	502	1460	500	1265	3.5	1.3	4.0	2.0
No. 18	Jan. 21, 1925	780	1560	570	1200	...	...	...	...
No. 3	Dec. 1, 1924	560	1230	338	630	3.0	2.5	5.5	3.5
(Corre	cted Results)								
No. 19	Dec. 10, 1924	600	1560	780	1290	3.0	2.0	3.0	2.0

In commenting on the demulsibility test, several members reported difficulty in obtaining readings when the test is run at 100° F., due to an indistinct line of demarcation between the oil and emulsion. One member points out that the effect of the viscosity of the oil on the test is greater at 100° F. than at 130° F., and that the latter temperature is nearer that encountered in use and is, therefore, more desirable for the emulsion test.

Two members favor the demulsibility test because of the simplicity of operation and the more perfect standardization of the apparatus.

One member suggests that the effect of the distilled water be determined in doing further work on the demulsibility test.

Mr. Phillips reports that the Sinclair test shows a greater difference between these two oils than the other two tests. Sample No. 24-1 shows "very good" as the oil layer is bright, water layer is clear, and there is only a faint trace of emulsion. Sample No. 24-2 is, however, of inferior quality; the oil layer is bright, water layer milky, and there is a $\frac{1}{16}$-in. layer of emulsion. These results were checked by the chairman's laboratory. Mr. Phillips is of the opinion that there are in reality but two kinds of turbine oils, (1) those which are entirely free of emulsifying properties, and (2) those which show emulsion formers of some kind. The first is the ideal turbine oil and a test is needed to distinguish this class of oils from all others. He points out the fact that emulsion tests change notoriously with age and suggests the need of research into the causes of this trouble and its prevention.

It has been observed in the chairman's laboratory that one of the causes of variations in readings in seconds, when testing good oils by the Steam Emulsion Test, is that part of the water tends to separate from the emulsion before the end of the steaming period. This partial breaking takes place to a greater extent on some tests on the same sample than on others, and so affects the results. A few experiments have been tried to prevent this separation and it has been found that raising the steaming bath temperature to from 100 to 105° F., instead of 67 to 78° F., gave satisfactory results. Another method of preventing this trouble is to raise the temperature of the emulsion to 190 to 195° F. more slowly than usual, taking at least 90 seconds, instead of 45 to 75 seconds as at present. The first procedure is the simpler, and it is suggested that the members try this modification of the test and send in their comments on it. The two samples which have been sent out may not give trouble due to this separation now, as the tests on the oils are poorer than when they were first sent out, but the points mentioned above are worth investigating further. The chairman believes that the difference obtained when measuring the sample at 200° F., as compared with the measurement at room temperature, is so small as not to warrant the change. It is also believed that further refinements in the A.S.T.M. Steam Emulsion Test should be tried and that a definite specification should be drawn for the thermometer used in the steaming tube.

The Institution of Petroleum Technologists of Great Britain has published the A.S.T.M. Steam Emulsion Test with a few minor changes as a standard method of test. They specify that the steam piping or steam delivery tube shall be exactly 2.5 mm. in inside diameter, instead of 2.3 to 2.7 mm. They specify that the temperature of the emulsifying bath shall be brought to 67° F. at the start of the test, instead of 67 to 78° F. They also give definite specifications for the oil container tube thermometer. The results are reported in minutes and half minutes as the "Demulsification Number, I.P.T. Method."

CONCLUSIONS

The following conclusions are evident from the data reported by the nineteen separate laboratories:

Considering the two oils, designated No. 24-1 and No. 24-2, respectively, oil No. 24-1 would be classed as better than oil No. 24-2 by either the Herschel, the A.S.T.M. Steam Emulsion Test, the Sinclair Test, or by the Funk Sludging Method. Considering the main reason for circulating these oils, which was to determine whether or not the Herschel and Steam Emulsion Tests would distinguish between different grades of turbine oil and the degree of reproducibility possible in different laboratories, it should be noted that both tests will distinguish between the two oils, but that the average variations in results between separate laboratories seem to be greater in the case of the Herschel Test.

The following interpretation has been made of the demulsibility results obtained by the committee members in running tests on the two samples of oil.

	Oil No. 24-1		Oil No. 24-2	
	100° F.	130°F	100° F.	130° F.
Average time for 20 cc. to separate out, minutes	1.81	0.85	2.43	1.08
Average deviation, minutes	0.580	0.153	0.596	0.389
Average deviation, per cent	32.0	18.0	24.5	36.0

It is felt, however, that a common basis of comparison could only be obtained if the time for complete separation in the demulsibility test were read to the nearest second.

In view of the desire to have an emulsion test covering as wide a range of oils as possible, and considering the many difficulties reported in the past in checking the Herschel Test, it is recommended that the A.S.T.M. Method of Test for Steam Emulsion of Lubricating Oils (D 157 – 23 T) be kept tentative for another year and that further refinements in technique be investigated in order to make it even more satisfactory as a means of distinguishing between different grades of turbine oils.

Respectfully submitted on behalf of the sub-committee,

JAMES T-B. BOWLES,
Chairman.

REPORT OF SUB-COMMITTEE XIII ON NEUTRALIZATION NUMBER AND SAPONIFICATION

In considering the Tentative Method of Test for Free Acid in Lubricants (D 188 - 24 T), the sub-committee has agreed that:

1. The title of the method should be changed, for the reason that the test is one of neutralization and not one to identify specifically certain compounds which are acidic in nature;

2. Methods of determining the alkali or mineral acid content of the oils should be included. Heretofore no tests have been included to indicate to the user of the method if he is obtaining a reaction due to mineral acids or alkalies or due to certain constituents having acid characteristics;

3. The exact procedure for standardizing the standard titrating solution should be specified, as this will eliminate a factor of error in the procedure.

Therefore, the sub-committee recommends: (*a*) the withdrawal of Tentative Method of Test for Free Acid in Lubricants (D 188 - 24 T);[1] and (*b*) the adoption of the proposed Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants.[2]

The sub-committee recommends further that the name be changed to the Sub-Committee on Neutralization Number and Saponification.

ELECTROMETRIC METHOD

Three laboratories reported that they had assembled the equipment for carrying tests by means of the electrometric method. Two reported difficulty and the other was able to get consistent readings.

The apparatus has been simplified considerably. The outstanding features of the revised set-up, which has worked satisfactorily, are:

1. Use of bright platinum electrodes and discontinuance of mercury contacts;
2. Simple form of "bridge";
3. Use of alcoholic potassium hydroxide instead of sodium ethylate;
4. Means of equalizing the potential of the platinum electrodes.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 898 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 315.

[2] See p. 722.—ED.

The chairman has for experimental purposes electrodes of tungsten (2 types), molybdenum and gold, but he cannot report progress on their suitability.

The proposed method is appended hereto for publication as information.

Additional work on the proposed method is required to determine:

1. If other metals may be used for electrodes such as gold, tungsten, molybdenum.

2. By laboratories equipped with the necessary electrical apparatus on

(*a*) Variation in resistance of "salt bridge" agar agar+KI, due to diameter and length.

(*b*) Overall resistance of internal circuit. (The above data will furnish necessary specifications for the resistance of the series coil, and the desired sensitivity of the galvanometer.)

Various laboratories have agreed to install the apparatus and proceed to work on the method. Further work is in progress on the following:

- 0.1*N* and 0.02*N* KOH (alcoholic solution) electrometric and present indicator:
- 0.1*N* and 0.02*N* KOH (aqueous solution) present indicator on varied oils respecting both color and neutralization number.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

APPENDIX

PROPOSED METHOD OF TEST

FOR

NEUTRALIZATION NUMBER OF PETROLEUM OIL, TITRATION BY ELECTROMETRIC METHOD

Electrical Theory.—When two similar metallic electrodes are immersed in solutions having the same common ion in different proportions, and the two solutions are joined by a non-metallic conductor (salt bridge) an electromotive force is set up which varies with the difference in concentration in the two solutions. This may be used to measure the concentration difference, by titrating one of the solutions, until its evolved ion concentration is the same as that in the other solution, as a result of which the electromotive force reaches zero.

General Outline of Method.—The weight of oil under test is dissolved in a solvent (a single phase solution resulting), to which is added the necessary amount of alcoholic potassium hydroxide, the end point of the titration being indicated electrically. Platinum electrodes, one in the beaker under test, the other immersed in a comparison cell, containing the solvent and a plug of agar agar plus KI, are connected to a suitable galvanometer. The agar agar plus KI acts as a conductivity bridge. The end point in the titration is indicated by the zero deflection of the galvanometer. The e. m. f. generated by the two electrodes has been decreased by the addition of the alcoholic potassium hydroxide, until the end point is indicated.

I. APPARATUS AND REAGENTS

1. The apparatus shall consist of the following:

(*a*) A galvanometer of suitable sensitivity.

NOTE.—Experimental work was run on a galvanometer having a resistance of 1095 ohms and a sensitivity of 41 megohms. The manufacturer's catalog indicates for this type a sensitivity 0.025 microampere per millimeter, a period 3 seconds, an external critical damping resistance, open circuit, and a resistance 1060 ohms.

(*b*) A resistance coil, unmounted, 10,000 ohms, 0.2 per cent accuracy.

(*c*) A single contact key. Single pole, double throw, switch.

(*d*) A 250-cc. beaker.

(*e*) A comparison cell, glass tube, approximately 4½ in. long, 1⅛ in. in diameter. This tube has a capacity of 50 cc. A large test tube will do. A hole approximately ⅜ in. in diameter shall be blown near the bottom, on the parallel side of the tubing.

(*f*) Platinum electrodes, approximately 1 cm. square, sealed into soft soda glass, and copper lead wire sealed to platinum. Electrical connection by means of mercury is to be avoided. Electrodes shall be made of commercial grade platinum, 1 cm. square, 0.15 mm. thick (see Fig. 1).

NOTE.—It is suggested that the platinum be purchased already cut, as indicated. The taper edge can be filed on in the laboratory and should approximate a 30-deg. angle.

The copper wire shall first be spot welded or welded on with hard silver solder. Glass tubing of suitable bore shall be heated until softened and the wire is pushed through until the platinum strip is well within the tubing. Heating should continue until an effective seal is made and can be aided by squeezing the glass while still soft between two pieces of wood. The glass should be very carefully annealed. The glass should not run over the enlarged surface of platinum.

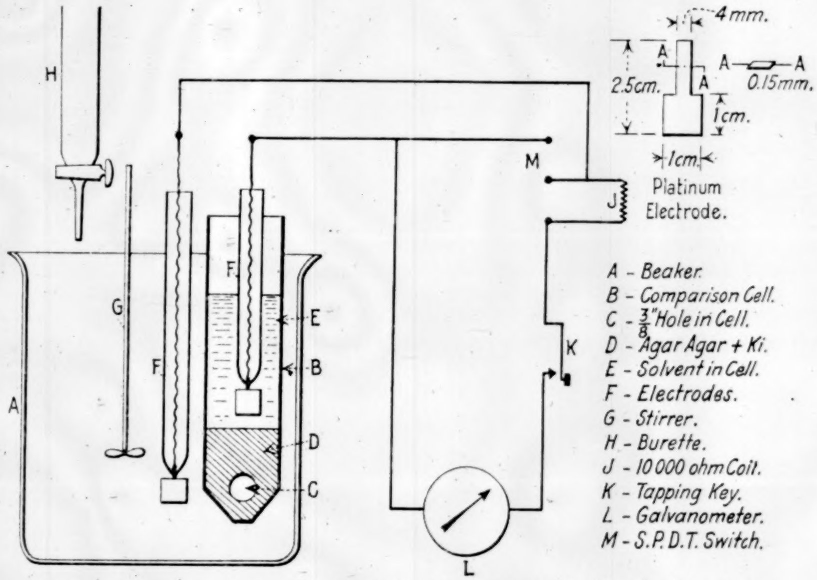
4 mm.
A
A
A—A
2.5cm.
0.15mm.
1cm.
1cm.
Platinum Electrode.
H
G
F
F
E
B
D
C
A
M
J
K
L
A - Beaker.
B - Comparison Cell.
C - $\frac{3}{8}$" Hole in Cell.
D - Agar Agar + Ki.
E - Solvent in Cell.
F - Electrodes.
G - Stirrer.
H - Burette.
J - 10 000 ohm Coil.
K - Tapping Key.
L - Galvanometer.
M - S.P.D.T. Switch.

FIG. 1.—Apparatus for Electrometric Titration Neutralization Number.

(*g*) Suitable mechanical stirrer, electric, air, or water motor.

2. The solutions shall consist of the following:

(*a*) *Solvent.*—Ten parts by volume of 90-per-cent water white benzol, 9 parts by volume of absolute or 95-per-cent alcohol (ethyl), 1 part by volume of saturated alcoholic potassium iodide.

NOTE.—The solvent should be neutral.

(*b*) *Agar Agar for comparison cell.*—Agar Agar, Merck's, 5 per cent maximum impurities, 1 g. agar agar boiled in 100 cc. of distilled water to which shall be added 10 cc. of a saturated alcoholic solution of potassium iodide.

(*c*) *Titrating Solution.*—Alcoholic potassium hydroxide, 1 cc. of which contains 5 mg. KOH.

This solution is to be standarized against Bureau of Standards certified benzoic acid.

1 cc. = 5 mg. KOH = 10.88 mg. benzoic acid.

The KOH solution may be conveniently standarized against benzoic acid, electrometrically. Refer to Bureau of Standards Certificate 39*b*, which accompanies the Standard Sample of benzoic acid.

II. PROCEDURE

3. Preliminary to making a determination, place both electrodes in a beaker containing sufficient solvent to cover the electrodes and short circuit the electrodes by joining the wire or closing the switch. The electrodes will in time equalize their potential, as will be shown by a zero galvanometer deflection. When the electrodes are not in use they should remain short circuited, immersed in the solvent.

A thoroughly clean tube is partly filled with the boiling agar agar jelly and allowed to congeal. Protect carefully from air exposure, when not in use.

After the oil sample, approximately 10 g., is weighed accurately, add to the beaker approximately 150 cc. of the solvent and add a sufficient amount of solvent to the comparison cell so that the platinum electrode may be sufficiently submerged. Insert the comparison cell in the beaker. Insert the other electrode in the beaker. Insert the stirrer.

It is important that the speed of agitation and the direction of rotation be such that no air bubbles are drawn into the solution.

From the burette add the KOH solution, permitting sufficient time between each addition for equilibrium to be established chemically.

The approach to the zero point is indicated by the decrease in the deflection and additions of KOH are made, until no deflection is noted. This is the end point.

Polarization must be avoided. The tapping key need be pressed only an instant. The resistance coil should be in series during all but the final stage of the titration, but near the end, the coil should be shunted out. Maximum sensitivity is required near the end point.

Burette readings indicate volume of alkali used, and the calculations give the neutralization number.

NOTE 1.—For optimum conditions, the galvanometer deflections should be as large as possible, particularly as the end point is approached, so that a sharp end point is obtained. To obtain such deflections, the galvanometer should be sensitive and the resistance of the entire circuit, including lead wires, solutions under test, comparison cell, should be kept low. The resistance of the solutions should be made as low as possible by adding potassium iodide to the solvent and by placing the electrodes as close as is consistent to the comparison cell.

NOTE 2.—Polarization of the electrodes should be kept to a minimum. Concentration cells polarize readily when continued currents are drawn, particularly if the current density is large. To minimize the polarization of the electrodes while under test, the tapping key should be depressed but an instant. The electrode area should be large. Another means of minimizing polarization is the introduction of a resistance coil in the external circuit to cut down current flow while the e. m. f. is large. When the end point is approached the resistance may be shunted out, in order to give a more sensitive end point.

REPORT OF SUB-COMMITTEE XV ON SAMPLING AND GAGING

The sub-committee has devoted its attention this year chiefly to the subject of an abridged table for correcting the volume of petroleum products to a 60° F. temperature base.

In conjunction with the Bureau of Standards and the chairman of the Advisory Committee of the American Petroleum Institute on Testing Methods and Specifications for Petroleum and Its Products, an agreement has been reached on the details of such a table and this proposed table is now submitted[1] for presentation to the Society as a standard on this subject.

It will be observed that this Abridged Table is divided into six groups with basic coefficients of expansion of 0.0004, 5, 6, 7, 8, 85. The multipliers in the various groups, except as noted in the table, are obtained directly from Table II of *Circular 154*, "National Standard Petroleum Oil Tables" of the Bureau of Standards, using the A. P. I. gravity which corresponds to the basic coefficients of expansion, rounding off to the nearest integer.

Respectfully submitted on behalf of the sub-committee,

C. N. FORREST,
Chairman

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[1] See A.S.T.M. Standards Adopted in 1925.—ED.

REPORT OF SUB-COMMITTEE XX ON NOMENCLATURE

In the report of the sub-committee for 1924 certain definitions were published for information and criticism. These definitions have been critically reviewed by the Executive Committee of the Standardization Committee of the Institution of Petroleum Technologists, and many helpful suggestions have been received which have enabled the sub-committee to so revise certain of these definitions that they may apply both to American and English practice. The sub-committee wishes to express its appreciation of this cooperation on the part of the Institution of Petroleum Technologists. The sub-committee has also received certain editorial suggestions from the Editorial Committee of Committee E-8 on Nomenclature and Definitions, some of which the sub-committee has adopted and incorporated in the definitions here presented. The sub-committee again requests criticisms and suggestions in the matter of the definitions submitted. Two additional definitions have been incorporated, namely for "engine distillate" and "tops," and the definition for "mineral seal oil" has been omitted pending further consideration of a proper designation for this product.

The sub-committee wishes to call particular attention to the spelling of the word "kerosine." This matter was brought to our attention by the Executive Committee of the Standardization Committee of the Institution of Petroleum Technologists, it being pointed out that the ending "ene" is very generally applied to pure hydrocarbons. The suffix "ine" already applied to gasoline is therefore also applied to kerosine.

Crude Petroleum.—A naturally occurring mixture, consisting predominantly of hydrocarbons, or of sulfur, nitrogen or oxygen derivatives of hydrocarbons, which is removed from the earth in liquid state or is capable of being so removed.

Note.—Crude petroleum is commonly accompanied by varying quantities of extraneous substances such as water, inorganic matter and gas. The removal of such extraneous substances alone does not change the status of the mixture as crude petroleum. If the removal of such extraneous substances also results in the removal of more than a trace of the naturally occurring mixture, the resulting product is no longer crude petroleum.

Topped Crude Petroleum.—A residual product remaining after the removal, by distillation, or other artificial means, of an appreciable quantity of the volatile components of crude petroleum.

Weathered Crude Petroleum.—The product resulting from crude petroleum through loss, due to natural causes, during storage and handling, of an appreciable quantity of the more volatile components.

Benzine.—This term is archaic and misleading and should not be used.

Petroleum Naphtha.—A generic term applied to refined or unrefined petroleum products and liquid products of natural gas, not less than 10 per cent of which distills below 347° F. (175° C.) and not less than 95 per cent of which distills below 464° F. (240° C.), when subjected to distillation in accordance with the current method of test of the American Society for Testing Materials for petroleum products of this nature.

NOTE.—The "naphthas" used for specific purposes such as cleaning, manufacture of rubber, manufacture of paints and varnishes, etc., are made to meet specifications which may require products of considerably greater volatility than that set by the maximum limits of this generic definition.

Gasoline.—A refined petroleum naphtha which by its composition is suitable for use as a carburant in internal combustion engines.

Petroleum Spirits—White Spirits.—A refined petroleum distillate, with a minimum flash point of 70° F. (21° C.) (Tag Closed Tester, A.S.T.M. Standard or Abel Tester) with volatility and other properties making it suitable as a thinner and solvent in paints, varnishes and similar products.

NOTE.—The term "turpentine substitute" as applied to petroleum spirits is to be condemned as false and misleading. The term "mineral spirits" also should not be used as it includes within its scope not only petroleum products, but other hydrocarbon mixtures, such as coal-tar distillates.

In Great Britain the term "petroleum spirits" is applied to a very light hydrocarbon mixture having a flash point below 32° F. (0° C.).

Kerosine.—A refined petroleum distillate having a flash point not below 73° F. (23° C.), as determined by the Abel Tester (which is approximately equivalent to 73° F. (23° C.) Tag Closed Tester, A.S.T.M. method) and suitable for use as an illuminant when burned in a wick lamp.

NOTE.—Local ordinances at times require flash points higher than 73° F. (23° C.). Tag Closed Tester, A.S.T.M. Standard.

Gas Oil.—A liquid petroleum distillate having a viscosity intermediate between that of kerosine and lubricating oil.

NOTE.—It should be understood that oils, other than gas oil as defined above, may be and are used in the manufacture of gas.

Fuel Oil.—Any liquid or liquefiable petroleum product used for the generation of heat in a furnace or firebox, the controlling factors being cost and adaptability. Fuel oils in common use fall into one of four classes: (*a*) Residual fuel oils, which are topped crudes or viscous residuums obtained in refining processes; (*b*) Distillate fuel oils, which are distillates derived directly or indirectly from crude petroleum, (*c*) Crude petroleums and weathered crudes of relatively low commercial value; (*d*) Blended fuels, which are mixtures of two or more of the three classes.

Tar, Pitch.—These terms shall not be applied to petroleum products.

Petroleum Lubricating Grease.—A combination of a petroleum product and a soap or a mixture of soaps, suitable for certain types of lubrication.

Oil-Shale.—A compact rock of sedimentary origin, yielding over 33 per cent of ash and containing organic matter that yields oil when destructively distilled but not appreciably when extracted with the ordinary solvents for petroleum.

Crude Shale Oil.—The oil obtained as a distillate by the destructive distillation of oil-shale.

End Point.—The highest temperature reading observed on the distillation thermometer in conducting the A.S.T.M. distillation test for gasoline, etc.

Engine Distillate.—A refined or unrefined petroleum distillate similar to naphtha, but often of higher distillation range.

Tops.—The unrefined distillate obtained in topping a crude petroleum.

Respectfully submitted on behalf of the sub-committee,

K. G. MACKENZIE,
Chairman.

REPORT OF SUB-COMMITTEE XXI ON CRUDE PETROLEUM

Work has been done on the naphtha content of three crude oils: Montebello crude, Mexia crude and a Mid-Continent crude. The apparatus used consisted essentially of a copper flask of two liters capacity, and an especially designed brass column, used in conjunction with the condenser specified in the Tentative Method of Test for Distillation of Gasoline (D 86 - 24 T).[1] A charge of 1000 cc. of crude was used, and the condenser temperature was maintained between 0 and 4.5° C. (32 and 40° F.). The rates of distillation were as follows:

Up to 75° C. (167° F.)	2.0 – 2.5 cc. per minute
75-100° C. (167-212° F.)	4.0 – 5.0 " " "
Above 100° C. (212° F.)	9.0 – 11.0 " " "

The slow initial rate was employed because of the use of the comparatively small condenser. The method of operation involved reading the volume of distillate at 392° F. (200° C.), and dividing by 0.9 to obtain the volume at which the cut is actually made. One hundred cubic centimeters of the clear distillate were measured out and distilled according to Method of Test D 86 – 24 T, except that instead of reading temperatures at each 10-per-cent point, the percentages distilling at 221° F. (105° C.), 284° F. (140° C.), and 392° F. (200° C.) were noted.

The results were calculated as follows:

$$\frac{C - D}{A - D} \times 100 = \text{percentage of naphtha (meeting the requirement of 90 per cent distilling at 392° F. (200° C.)).}$$

where A = the initial charge,
C = the total volume of the naphtha cut, separated in the distillation of the crude, and
D = the volume of water collected in the receiver.

The results of this work are given in the accompanying Table I. It will be noted that satisfactory checks are obtained for the percentages of naphtha in the crudes. The agreement in the percentages reported as distilling at 221° F. (105° C.) and 284° F. (140° C.) is not very satisfactory.

It has been agreed upon that the work of the immediate future shall consist of a study of the method with the charge of crude 1000 cc., capacity of still 3000 cc., and the length of column 35.6 cm. (14 in.) The regular A.S.T.M. condenser shall be used. Below 212° F. (100° C.) the rate shall be 5 cc. per minute, and the temperature of the condenser water shall be maintained between 10 and 14° F. (−12 and −10° C.) by the use of salt and ice. Above 212° F. (100° C.), the rate of distillation may be increased to 10 cc. per minute, and the temperature in the condenser shall be maintained at 32 to 40° F. (0 to 4.5° C.).

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 864 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 258.

TABLE I.—RESULTS OF TESTS TO DETERMINE NAPHTHA CONTENT OF CRUDE OILS.

Operator	Mexia Crude						Montebello Crude						Mid-Continent Crude					
	Percentage of Naphtha	F. D.	Per Cent at 221° F.	Per Cent at 284° F.	Per Cent at 392° F.	End Point, deg. Fahr.	Percentage of Naphtha	F. D.	Per Cent at 221° F.	Per Cent at 284° F.	Per Cent at 392° F.	End Point, deg. Fahr.	Percentage of Naphtha	F. D.	Per Cent at 221° F.	Per Cent at 284° F.	Per Cent at 392° F.	End Point, deg. Fahr.
1[a]........	20.7	164	7.0	36.0	93.0	430	15.4	200	2.0	30.0	91.5	436	35.7	131	22.0	52.0	94.0	437
	21.5	170	7.0	35.0	92.0	437	15.5	204	1.0	29.0	89.0	438	....	...	....	....	....	...
2........	22.2	149	11.0	35.0	93.0	415	15.7	174	4.5	35.0	94.0	417	37.7	102	25.0	52.0	94.0	414
	22.2	149	11.0	36.0	94.0	412	15.9	172	4.0	35.0	94.0	419	38.1	108	24.0	51.0	93.0	419
3........	22.9	129	11.0	34.0	87.5	435	15.7	174	4.5	38.0	91.5	424	37.6	104	25.0	56.0	93.0	424
	22.7	124	12.0	33.0	88.0	430	15.9	172	5.0	38.0	92.0	426	37.4	103	25.0	56.0	92.5	423
4........	22.4	149	7.5	33.0	92.0	419	15.3	212	0.5	33.5	93.5	419	37.0	122	20.0	50.0	92.5	421
	22.2	145	8.0	35.0	93.0	419	....	...	....	....	....	...	....	...	....	....	....	...
5........	22.8	120	10.2	36.0	93.9	416	15.6[b]	180	3.0	35.0	93.5	420	37.9	111	21.4	51.4	92.6	422
	22.9	124	10.0	35.9	94.0	416	....	...	....	....	....	...	37.8	110	21.7	51.0	92.2	423
6........	22.8	180	6.0	31.0	93.0	418	16.2	210	1.5	31.0	92.0	420	36.1	148	16.5	49.0	94.0	420
	23.1	176	6.0	31.0	94.0	420	16.1	212	1.0	31.0	93.0	419	36.1	152	16.5	49.5	94.0	424
	23.0	178	5.5	30.5	94.0	415	16.0	210	1.0	30.0	92.0	424	35.9	220	16.0	49.0	93.5	422
7........	22.9	127	11.3	33.8	92.0	422	16.1	192	2.1	29.0	90.8	425	37.9	108	22.8	51.8	92.0	426
	23.4	126	11.1	35.0	92.7	420	16.3	188	2.8	31.0	91.3	420	37.8	110	22.2	50.0	92.3	424
8........	22.5	150	12.0	34.0	95.0	418	16.4	185	5.0	38.0	97.0	410	37.5	122	23.0	57.0	97.0	411
	22.3	150	12.0	34.0	95.0	423	16.5	189	5.0	38.0	97.0	411	37.4	122	23.0	58.0	98.0	410
9..........	23.0[b]	162	7.4	32.4	91.0	430	16.7[b]	199	2.8	30.3	92.6	425	38.1[b]	111	21.7	49.8	92.8	424
10..........	23.9	140	9.0	31.0	92.0	428	16.1	188	2.0	30.0	93.0	425	37.5	108	23.0	52.0	94.0	420

[a] Did not use regular method or apparatus.
[b] Average of three runs.

A sub-committee consisting of Messrs. Campbell, Haylett and Terry will undertake to develop a more permanent type of still, using the present still of the Associated Oil Co. as a basis, and proceeding along the line of suggestion given above.

A second sub-committee consisting of Messrs. Dean and Parsons will endeavor to work out a satisfactory method of calculating and reporting results.

Respectfully submitted on behalf of the sub-committee,

N. A. C. SMITH,
Chairman.

REPORT OF SUB-COMMITTEE XXIV ON CALORIFIC VALUE OF FUEL OIL

The sub-committee drew up a proposed tentative procedure for calorific value of fuel oil based on the standard method for the determination of the thermal value of coal appearing in the Standard Methods of Laboratory Sampling and Analysis of Coal (D 22 - 24).[1] Four samples of oil: a medicinal oil; a low-viscosity, low-sulfur fuel oil; a California oil; and a Mexican fuel oil were circulated and the following results in B.t.u. per lb. were secured:

	Low Viscosity Low Sulfur	Medicinal Oil	California	Mexican
Average	19 738	19 845	19 182	18 284
High	19 930	19 915	19 382	18 439
Low	18 877	19 643	19 009	18 047

With possibly two or three exceptions, cooperating members deviated from the proposed method, and in only a few instances were all the variables given. The sub-committee recommends that the method be not yet proposed as tentative, that more samples of oil be circulated for further work, that a complete report form be sent each member in order that all necessary data on exact procedures followed be obtained, and that the following changes be made in the proposed method:

1. Change the sentence under Oxygen in the Standard Methods D 22 - 24 now reading: "The bomb, when filled, shall contain at least 5 per cent of nitrogen to insure complete oxidation of the sulfur" to read "The bomb, when filled, shall contain at least 3 to 5 per cent of nitrogen, etc.";
2. Add an explanatory clause to show that no additional nitrogen is needed beyond that supplied by the air originally present in the bomb;
3. Add a sentence pointing out the necessity for analysis of the oxygen used; and
4. If later considered necessary, the inclusion of a suitable method for the analysis of the oxygen.

Respectfully submitted on behalf of the sub-committee,

F. A. Hull,
Chairman.

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[1] 1924 Book of A.S.T.M. Standards.

REPORT OF COMMITTEE D-4

ON

ROAD AND PAVING MATERIALS

Committee D-4 has held three meetings during the past year. One new method of test has been prepared and a number of existing tentative and standard specifications and methods of test have been revised.

Sections of the Sub-Committee on Bituminous Materials have been actively engaged during the year on new methods of test, but were unable to complete their investigations sufficiently early to enable the recommendation of tentative standards at the 1925 Annual Meeting of the Society. The test for so-called asphalt content or residue on evaporation of road oils under standard conditions, standard methods of inspecting and testing bituminous mixtures, and a standard method for testing emulsions have been under development and are expected to be in form for presentation during the coming year. The Section on Distillation is actively engaged in cooperative testing looking toward the adoption of a standard apparatus and method which will harmonize differences at present existing between at least three standing committees.

Sections of the Sub-Committee on Non-Bituminous Materials have also been active. It is believed that next year a modification of the standard abrasion test will be recommended to the Society and that considerable progress will be made in simplifying and harmonizing specifications for crushed stone, slag and gravel for use as road materials.

A special committee was appointed to confer with the chairman of the Sub-Committee on Definitions for Specific Gravity of Committee E-8 on Nomenclature and Definitions with the idea of harmonizing existing differences of opinion with respect to these definitions. Certain slight modifications in the definitions of Committee E-8 were worked out which if acceptable to that committee have the approval of Committee D-4 as ascertained by letter ballot which showed 36 members voting affirmatively, 0 negatively, and 31 who refrained from voting.

PROPOSED REVISIONS OF EXISTING STANDARD METHODS OF TEST

NO. 1. *Standard Method of Test for Penetration of Bituminous Materials (D 5 - 21).*[1]—In cooperation with the Bureau of Standards the Sectional Committee on Penetration has worked out a modification of the requirements for a standard penetration needle. It is believed these modifications will greatly facilitate the manufacture of these needles, which has caused considerable trouble in the past. A detailed report on this subject by the sectional committee is appended hereto. The following revisions are now recommended in this method:

Section 3.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets. This involves the revision of Fig. 1 to conform to the accompanying Fig. 1:

"The needle (Fig. 1) for this test shall be *made from* a cylindrical steel rod *approximately* 50.8 mm. (2 in.) long, *and* having a diameter of [1.01] *1.00* to 1.02 mm. [and having a taper of 6.34 to 6.36 mm. measured on the axis.] *This shall be symmetrically tapered at one end to a cone approximately 6.35 mm.* ($\frac{1}{4}$ *in.*) *in height and whose angle shall be within the range of 8° 40′ and 9° 40′.* After tapering, the point shall be "blunted" by grinding off to a truncated cone, the smaller base of which shall be from 0.14 to 0.16 mm. in diameter. *The finished needle shall be hardened and highly polished.*

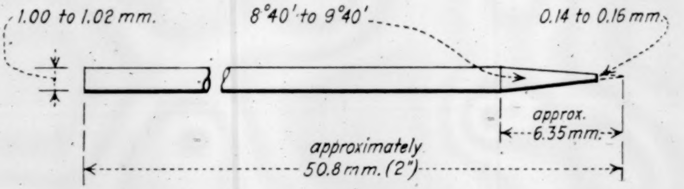

FIG. 1.—Penetration Needle.

Add a note to read as follows:

"NOTE.—The U. S. Bureau of Standards will measure and certify the accuracy of penetration needles in accordance with the tolerances given."

It is recommended that these revisions be incorporated in the present standard method without publication as tentative. The committee accordingly asks for the necessary nine-tenths vote for adoption of this revision as standard immediately.

NO. 2. *Standard Methods of Test for Specific Gravity of Sand, Stone and Slag Screenings, and Other Fine Non-Bituminous Highway Materials (D 55 - 24)*[1]—In order to make the Le Chatelier method described in this standard applicable to materials having a specific gravity lower than 2.67 and to allow for the use of water in making the test the following revisions are now recommended in this method.

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[1] 1924 Book of A.S.T.M. Standards.

Section 2.—Change the last sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> "Kerosene *or* [free from] water [or benzine not lighter than 62° Baumé,] *may* [shall] be used in making this determination."

Section 3 (a).—Change the first paragraph to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> "The flask shall be filled with either of these liquids to a point on the stem between zero and one cubic centimeter and *a reading taken. From 50 to* 64 g. (*depending upon its approximate specific gravity and to bring the reading on the scale*) of sand or other fine non-bituminous highway material of the same temperature as the liquid, shall be slowly introduced, taking care that the material does not adhere to the inside of the flask above the liquid and to free the material from air by rolling the flask in an inclined position. After all material is introduced, the level of the liquid *in the graduated stem shall be read and* [will rise to some division of the graduated neck;] the difference between readings is the volume displaced by the *known weight* [64 g.] of the material."

It is recommended that these revisions be incorporated in the present standard method, without publication as tentative. The committee accordingly asks for the necessary nine-tenths vote for adoption of the revision as standard immediately.

No. 3. *Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment* (*D 20 - 18*).[1]—The following changes are recommended in this method as tending to improve its accuracy and reliability.

Section 4.—Change the first sentence to read as follows by the addition of the italicized word and the omission of the word in brackets:

> "The apparatus shall be set up as shown in Fig. 2, the thermometer being placed so that the top of the bulb is opposite the *bottom* [middle] of the tubulature."

Make the position of the thermometer in the illustration correspond with this change.

Section 5.—Change the second sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> "After adjusting the thermometer, shield, condenser, etc., the distillation is commenced, the rate being so regulated that *50 to 70 drops* (*2.5 to 3.5 cc.*) *pass* [1 cc. passes] over every minute."

It is recommended that these revisions be accepted and published as tentative.

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[1] 1924 Book of A.S.T.M. Standards.

PROPOSED REVISIONS OF TENTATIVE SPECIFICATIONS AND METHODS OF TEST

No. 1. *Tentative Specifications for High-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product) (D 106 – 23 T)*.[1]—The following revision is recommended in these specifications:

Section 1 (c).—Change the first requirement relating to distillation to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"Total distillate, by weight, 0 to 170° C. (32 to 338° F.).......... *2.00 to 8.00 per cent* [3.00 to 10.00 per cent.]"

It is recommended that these specifications as revised be continued as tentative.

No. 2. *Tentative Specifications for High-Carbon Tar Cement (D 110 – 23 T)*.[2]—The following revision is recommended in these specifications:

Section 2 (c).—Omit the second requirement relating to distillation reading as follows:

"Total distillate, by weight, 0 to 235° C. (32 to 455° F.)..........not more than 2.00 per cent."

It is recommended that these specifications as revised be continued as tentative.

No. 3. *Tentative Method of Float Test for Bituminous Materials (D 139 – 24 T)*.[3]—The following changes are recommended in this method as tending to improve its accuracy and reliability:

Section 1.—Omit the last line of the table of requirements, reading as follows:

"Height of rim above water with load of 5.5 g., mm........7.0 min., 8.5 normal, 10.0 max."

Section 3.—Insert a new Section 3 reading as follows, renumbering the subsequent sections accordingly.

"3. *Assembly*.—The assembled float and collar, with the collar filled flush with the bottom and weighted to a total weight of 53.2 g., shall float upon water with the rim 8.5 ± 1.5 mm. above the surface of the water. Dimensions of the apparatus additional to those required above are given in Fig. 1."

Section 4.—Change from its present form: namely,

"The minimum diameter of the bath shall be twice the diameter of the float; the minimum depth of water shall be equal to the diameter of the bath."

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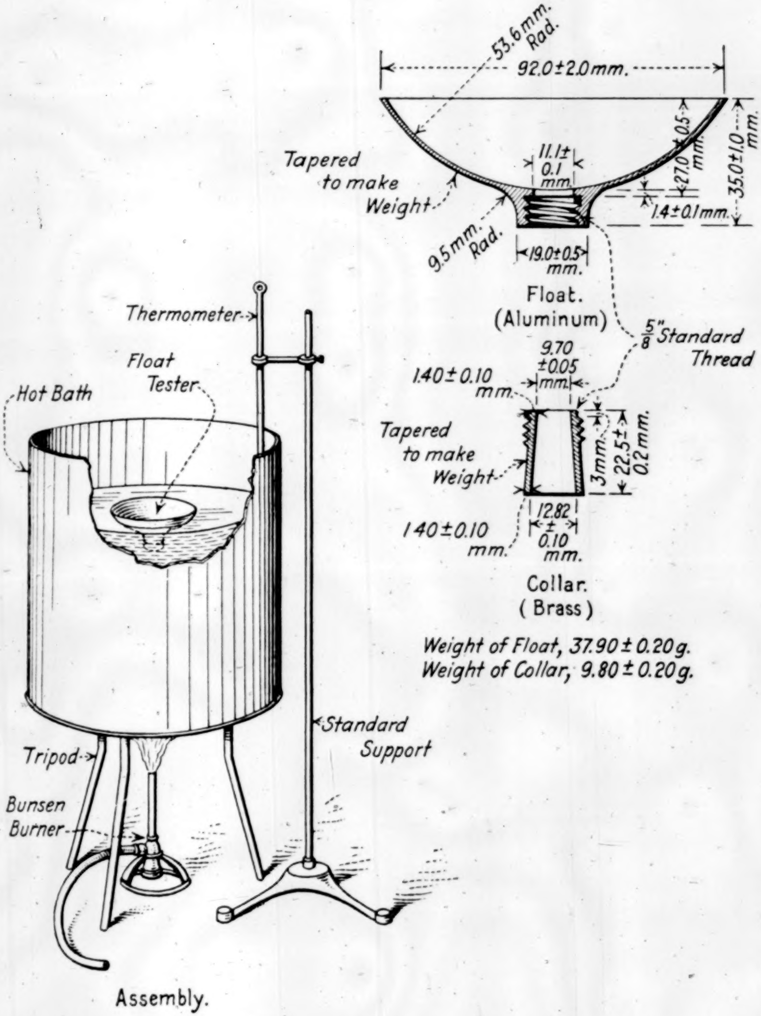
[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 735 (1923); also 1924 Book of A.S.T.M. Tentative Standards, p. 373.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 743 (1923); also 1924 Book of Tentative Standards, p. 381.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 925 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 407.

to read as follows:

"The diameter of the bath and the depth of water shall be at least 185 mm."



53.6 mm. Rad.
92.0±2.0 mm.
Tapered to make Weight
11.1± 0.1 mm.
27.0±0.5 mm.
35.0±1.0 mm.
1.4±0.1 mm.
9.5 mm. Rad.
19.0±0.5 mm.
Float. (Aluminum)
5/8" Standard Thread
9.70 ±0.05 mm.
1.40±0.10 mm.
Tapered to make Weight
3 mm.
22.5±0.2 mm.
12.82 ± 0.10 mm.
1.40±0.10 mm.
Collar. (Brass)
Weight of Float, 37.90±0.20 g.
Weight of Collar, 9.80±0.20 g.
Thermometer
Float Tester
Hot Bath
Standard Support
Tripod
Bunsen Burner
Assembly.

FIG. 2.

Section 5.—Reverse the position of the first two paragraphs and change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"The brass collar shall be placed with the smaller end on a brass plate, which has been previously amalgamated with mercury by first rubbing it with a dilute solution of mercuric chloride or nitrate, and then with mercury. [The sample shall be poured into the collar in any convenient way until slightly more than level with the top.]

"The sample shall be completely melted at the lowest possible temperature *that will bring it to a sufficiently fluid condition for pouring, excepting creosote oil residues, which shall be mixed and poured at a temperature of 100 to 125° C.* [heating only sufficiently to bring the product into a fluid condition but not above 350° F.,] *It shall be* [and] stirred thoroughly until it is homogeneous and free from air bubbles. *The sample shall then be poured into the collar in any convenient manner until slightly more than level with the top.*"

Change the last paragraph of this section to read as follows by the omission of the words and figures in brackets:

"*Tar Products.*—Tar products shall be [poured at a temperature of 100 to 125° C. and] immediately immersed in ice water maintained, etc."

Figure 1.—Change Fig. 1 to conform to the accompanying Fig. 2.

It is recommended that this method as revised be continued as tentative.

No. 4. *Tentative Method of Mechanical Analysis of Subgrade Soils (D 137 - 24 T).*[1]—The following changes are recommended in this method:

Section 2.—Change the last sentence to read as follows by the addition of the italicized words and figures:

"The sample shall then be passed through a ¼-in. screen and the material passing this screen further pulverized by rolling out in a thin layer on a rubber pad, using a rubber-covered roller, *after which the sample shall be passed through a 10-mesh sieve.*"

Section 4.—Change the first sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"By the method of quartering, a sample weighing approximately 25 g. *and passing the 10-mesh sieve shall be selected and* [prepared as specified in Section 2, shall be] placed in a beaker with approximately 500 cc. of distilled water."

It is recommended that this method as revised be continued as tentative.

No. 5. *Tentative Method of Test for Consistency of Portland-Cemen Concrete for Pavements or for Pavement Base (D 138 - 22 T).*[2]—The following changes are recommended in this method:

Title.—Change the title to read as follows. This change has the approval of Committee C-9 on Concrete and Concrete Aggregates.

"Tentative Method of Test for Consistency of Portland-Cement Concrete."

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 922 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 395.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 805 (1922); also 1924 Book of A.S.T.M. Tentative Standards, p. 398.

Section 4.—Change the next to the last sentence to read as follows by the addition of the italicized word and the omission of those in brackets:

> "The mold shall then be removed by being raised vertically, *immediately* [and at exactly three minutes] after being filled."

It is recommended that this method as revised be continued as tentative.

New Method of Test Submitted as Tentative

The committee recommends for publication as tentative the following method of test appended hereto:[1]

Method of Test for the Determination of Moisture Equivalent of Subgrade Soils in the Field.

Tentative Methods of Sampling Advanced to Standard

The committee recommends that the following tentative methods of sampling be advanced to standard:

Tentative Methods of Sampling Bituminous Materials (D 140 – 23 T).[2]

The results of the letter ballot of the committee upon the recommendations made in this report are as follows:

Items	Affirmative	Negative	Not Voting
I. Proposed Revision of Existing Standards			
Standard Method of Test for Penetration of Bituminous Materials (D 5 – 21)	39	0	28
Standard Methods of Test for Specific Gravity of Sand, Stone and Slag Screenings and Other Fine Non-Bituminous Highway Materials (D 55 – 24)	39	2	26
Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (D 20 – 18)	37	0	30
II. Proposed Revisions of Existing Tentative Standards			
Tentative Specifications for High-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product) (D 106 – 23 T)	29	2	36
Tentative Specifications for High-Carbon Tar Cement (D 110–23 T)	30	1	36
Tentative Method of Float Test for Bituminous Materials (D 139 – 24 T)	35	2	30
Tentative Method of Mechanical Analysis of Subgrade Soils (D 137 – 24 T)	32	1	34
Tentative Method of Test for Consistency of Portland Cement Concrete for Pavements or for Pavement Base (D 138 – 22 T)	34	1	32
III. Proposed New Tentative Standard			
Field Method for Determining the Moisture Equivalent of Subgrade Soils	29	2	36
IV. Advancement of Tentative Standard to Standard			
Tentative Methods of Sampling Bituminous Materials (D 140 – 23 T)	36	1	30

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[1] See p. 745.—Ed.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 759 (1923); also 1924 Book of A.S.T.M. Tentative Standards, p. 411.

This report has been submitted to letter ballot of the committee, which consists of 67 members, of whom 41 have voted affirmatively, 1 negatively, and 25 have refrained from voting.

Respectfully submitted on behalf of the committee,

H. S. MATTIMORE,
Chairman.

PRÉVOST HUBBARD,
Secretary.

EDITORIAL NOTE

The proposed revisions of the Standard Method of Test for Penetration of Bituminous Material; and of the Standard Methods of Test for Specific Gravity of Sand, Stone and Slag Screenings and Other Fine Non-Bituminous Highway Materials recommended for the first time in 1925, were approved at the annual meeting by a nine-tenths vote and subsequently adopted as standard by letter ballot of the Society on August 28, 1925. The methods as revised appear in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

The proposed revisions of the Standard Method of Test for Distillation of Bituminous Material Suitable for Road Treatment were accepted for publication as tentative and appear on page 911.

The Tentative Method of Sampling Bituminous Materials were approved at the annual meeting and subsequently adopted by letter ballot of the Society on August 28, 1925, and appear in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

The proposed revisions of the Tentative Specifications for High-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product); and for High-Carbon Tar Cement; and of the Tentative Method of Float Test for Bituminous Materials; of the Tentative Method of Mechanical Analysis of Subgrade Soils; and of the Method of Test for Consistency of Portland Cement Concrete for Pavements or for Pavement Base were accepted. The tentative specifications and methods as thus revised appear on pages 738, 740, 748, 742, and 746, respectively.

The proposed new Method of Test for the Determination of Moisture Equivalent of Subgrade Soils in the Field was accepted for publication as tentative and appears on page 745.

APPENDIX

REPORT OF SECTIONAL COMMITTEE ON PENETRATION

Reviewing, briefly, the earlier activities of the sectional committee, just prior to the annual meeting of 1923 the U. S. Bureau of Standards had drawn the Society's attention to certain features of the present needle requirements which seemed to warrant revision. These were the possibly undue restrictions as to the height of conical section which, together with the body diameter, allows an angle variation of only 7 minutes and also the impracticable feature of having to measure to an imaginary point in calibrating needles for certification.

The Bureau was of the opinion that if the requirements could perhaps be liberalized, it would greatly facilitate the procurement of specification needles. In this connection it was suggested that, instead of specifying the length of the conical section, we specify the included angle of the cone. It was also suggested that a 1-deg. range might be found satisfactory but the Bureau offered to cooperate by making either needles or tests in arriving at proper tolerances for such a feature.

As reported at the meeting of March 11, 1924, the sectional committee was of the opinion that specification needles were obtainable, but the importance of the Bureau of Standards' suggestions and its activities in certifying needles were emphasized. It was accordingly decided to accept its offer of assistance and they were invited to submit needles of suggested maximum and minimum angle tolerances together with their penetration tests for same.

In September, 1924, these needles were received by the chairman together with a report from which the following descriptions are quoted.

	Angle of Cone	Diameter of Point	Diameter of Body
A.S.T.M. Standard Needle[1]........	9 deg. 2 min.	......	0.0400 in. (1.016 mm.)[a]
Bureau of Standards Needle No. 1 (L4492-1)....	9 deg. 42 min.	0.0063 in. (0.16 mm.)[a]	0.0400 in. (1.016 mm.)[a]
Bureau of Standards Needle No. 2 (L4492-2)....	8 deg. 39 min.	0.0055 in. (0.14 mm.)[a]	0.0401 in. (1.018 mm.)[a]

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[1] Standard Method of Test for Penetration of Bituminous Materials (D 5 - 21), 1924 Book of A.S.T.M. Standards.

[a] Metric equivalents calculated by the chairman.

TABLE I.—RESULTS OF TESTS BY VARIOUS INVESTIGATORS ON PENETRATION OF BITUMINOUS MATERIALS.

		Bureau of Standards Needle No. 1	Bureau of Standards Needle No. 2	A.S.T.M. Standard Needle
SUBMITTED BY HERBERT SPENCER, STANDARD OIL CO. OF NEW JERSEY				
Standard Heavy Flux		179	181	181
New York Binder		132	134	133
51 to 60 Penetration Paving Asphalt		53	55	55
175 Oxidized Asphalt		23	23	24
300 Oxidized Asphalt		6	6	6
SUBMITTED BY C. S. REEVE, THE BARRETT CO.				
Steam-Refined Asphalt		116.5 119.0 118.0	121.5 120.5 118.0	122.5 119.0 120.5
	Average	117.8	120.0	120.7
Steam-Refined Asphalt		39.5 37.0 38.0	41.0 40.5 39.0	40.0 40.0 38.5
	Average	38.2	40.2	39.5
Blown Asphalt		73.5 75.0 74.0	76.5 78.5 76.5	77.5 77.0 77.0
	Average	74.2	77.2	77.2
SUBMITTED BY A. W. DOW, DOW AND SMITH[a]				
No. 1. Straight Steam Distillation	Low	99	100	99
	High	101	101	101
	Average	100	101	100
No. 2 Straight Steam Distillation	Low	128	130	126
	High	131	132	128
	Average	130	131	127
No. 3. Straight Steam Distillation	Low	146	146	142
	High	148	147	144
	Average	147	147	143
No. 4. Blown Asphalt	Low	84	86	88
	High	86	86	88
	Average	85	86	88
No. 5. Blown Asphalt	Low	102	104	103
	High	103	104	104
	Average	102	104	103
No. 6. Blown Asphalt	Low	124	124	121
	High	126	125	123
	Average	125	125	123

[a] Five penetration tests were made on each sample. The results reported state the highest and lowest and average. The A.S.T.M. Standard needle used had the following measurements:

Length of taper to theoretical point	6.25 mm.
Diameter of needle	1.021 mm.
Diameter of needle at point	0.1574 mm.

TABLE I.—RESULTS OF TESTS BY VARIOUS INVESTIGATORS ON PENETRATION OF BITUMINOUS MATERIALS (*Continued*).

		Bureau of Standards		A.S.T.M. Standard				
		Needle No. 1	Needle No. 2	Needle No. 1	Needle No. 2	Needle No. 3	Needle No. 4	Needle No. 5
SUBMITTED BY J. S. MILLER, THE BARBER ASPHALT CO.								
Oxidized Asphalt 15 to 20 Penetration at 77° F., 100 g., 5 sec............	Operator G	20	20	19	20	19	19	19
		19	21	20	20	20	19	19
		20	20	20	20	20	19	19
	Operator M	20	20	20	20	20	20	20
		20	20	20	20	20	20	20
		20	20	20	20	20	20	20
Oxidized Asphalt, 30 to 40 Penetration at 77° F., 100 g., 5 sec............	Operator G	34	35	35	36	36	37	36
		35	36	35	36	36	37	36
		35	36	35	36	36	36	36
	Operator M	36	37	37	37	37	36	36
		36	37	36	37	37	37	36
		36	37	36	36	37	36	36
Steam-Refined Asphalt, 30 to 40 Penetration at 77° F., 100 g., 5 sec.........	Operator G	33	34	34	34	34	34	34
		33	35	34	34	34	34	34
		33	34	34	34	34	34	34
	Operator M	34	34	34	34	34	34	33
		34	35	35	34	34	34	33
		34	35	34	34	34	34	33
Steam-Refined Asphalt, 50 to 60 Penetration at 77° F., 100 g., 5 sec.........	Operator G	57	58	56	57	57	57	57
		56	57	57	57	57	57	57
		57	57	57	56	57	57	57
	Operator M	56	58	57	57	57	57	58
		57	59	58	58	57	58	57
		56	59	57	58	57	57	57
Steam-Refined Asphalt, 85 to 100 Penetration at 77° F., 100 g., 5 sec.........	Operator G	88	90	89	88	88	88	88
		88	90	88	88	88	89	88
		87	90	88	89	88	89	88
	Operator M	89	91	90	89	89	89	89
		88	92	90	88	90	90	89
		89	90	90	88	89	90	89
Trinidad Asphalt Cement, 50 to 60 Penetration at 77° F., 100 g., 5 sec.....	Operator G	56	57	56	57	57	56	57
		55	57	57	57	56	57	56
		56	57	55	57	56	57	56
	Operator M	55	57	56	55	57	56	55
		56	57	56	55	56	57	55
		54	58	55	56	56	58	56

	Bureau of Standards Needle No. 1	Bureau of Standards Needle No. 2	A.S.T.M. Standard Needle
SUBMITTED BY HENRY M. MILBURN, U. S. BUREAU OF PUBLIC ROADS			
Sample No. 1..	19	22	22
	19	22	21
	19	22	22
	Average 19	22	21.6
Sample No. 2..	50	51	52
	50	50	52
	50	50	52
	Average 50	50.3	52
Sample No. 3..	92	92	93
	92	91	93
	93	92	93
	Average 92.3	91.6	93

TABLE I.—RESULTS OF TESTS BY VARIOUS INVESTIGATORS ON PENETRATION OF BITUMINOUS MATERIALS (*Continued*).

		Bureau of Standards Needle No. 1	Bureau of Standards Needle No. 2	A.S.T.M. Standard Needle
SUBMITTED BY J. E. MYERS, STATE HIGHWAY DEPARTMENT, ALBANY, N. Y.				
Sample No. 1		51	49	54
		50	50	52
		50	50	53
		Average 50	50	53
Sample No. 2		103	99	104
		100	102	102
		100	98	106
		Average 100	100	104
Sample No. 3		133	131	137
		134	134	137
		132	131	138
		Average 133	132	137
SUBMITTED BY THE NEW ORLEANS REFINING CO., INC., NEW ORLEANS, LA.				
Oxidized Asphalt, 10 to 15 Penetration	Operator D	11	12	10
		11	11	10
		10	11	10
	Operator L	10	12	9
		11	12	10
		11	12	11
Oxidized Asphalt, 30 to 40 Penetration	Operator D	33	33	34
		33	33	34
		33	35	33
	Operator L	35	35	36
		35	35	35
		33	36	36
Steam-Refined Asphalt, 30 to 40 Penetration	Operator D	33	34	32
		33	34	34
		33	34	34
	Operator L	35	35	36
		34	36	36
		34	37	36
Steam-Refined Asphalt, 50 to 60 Penetration	Operator D	51	51	50
		51	50	50
		51	50	50
	Operator L	52	51	53
		52	51	52
		52	52	51
Steam-Refined Asphalt, 85 to 100 Penetration	Operator D	82	81	79
		84	83	80
		83	83	79
	Operator L	83	86	82
		84	85	82
		84	84	84

TABLE I.—RESULTS OF TESTS BY VARIOUS INVESTIGATORS ON PENETRATION OF BITUMINOUS MATERIALS (*Continued*).

		Bureau of Standards Needle No. 1	Bureau of Standards Needle No. 2	A.S.T.M. Standard Needle
SUBMITTED BY U. S. BUREAU OF STANDARDS				
No. 1. Asphalt, 20 to 30 Penetration (Determination A)		26.0	28.0	28.0
		25.0	28.0	28.5
		25.0	26.0	28.0
		26.0	28.0	28.5
		27.0	28.0	27.5
		26.5	27.0	28.0
		26.5	27.0	27.0
		26.0	28.0	27.5
		25.0	28.0	28.5
		26.0	27.0	28.0
	Average	25.9	27.7	28.0
	Maximum	27.0	28.0	28.5
	Minimum	25.0	26.0	27.0
No. 1. Asphalt, 20 to 30 Penetration (Determination B)		27.0	27.0	26.0
		26.0	29.0	28.0
		27.0	28.0	28.0
		26.0	27.5	27.0
		27.0	28.0	28.0
		26.5	28.5	27.5
		27.5	28.0	28.0
		26.5	28.5	27.5
		27.0	28.0	27.5
		26.5	27.5	28.0
	Average	26.7	28.1	27.6
	Maximum	27.5	29.0	28.0
	Minimum	26.0	27.0	26.0
No. 2. Asphalt, 50 Penetration (Determination A)		52.0	53.0	52.0
		50.0	52.5	50.0
		51.5	51.5	52.0
		50.5	50.5	50.0
		51.5	52.0	51.0
		51.0	52.5	53.0
		50.0	53.0	50.0
		50.5	52.5	52.0
		51.0	53.0	52.0
		51.5	51.5	51.0
	Average	51.0	52.2	51.3
	Maximum	52.0	53.0	53.0
	Minimum	50.0	50.0	50.0
No. 2. Asphalt, 50 Penetration (Determination B)		49.0	52.5	52.0
		50.0	53.5	53.0
		49.0	53.5	53.5
		49.5	53.0	52.5
		51.0	53.5	53.5
		51.5	54.0	53.0
		49.5	53.5	52.0
		50.0	53.5	52.5
		50.0	53.5	52.0
		49.5	53.0	51.5
	Average	49.9	53.4	52.6
	Maximum	51.5	54.0	53.5
	Minimum	49.0	52.5	51.5
No. 3. Asphalt, 80 to 90 Penetration (Determination A)		81.0	84.0	83.0
		82.5	85.0	81.5
		83.0	85.0	81.0
		80.0	83.5	82.0
		81.5	86.0	83.0
		84.0	85.0	81.0
		82.5	84.0	82.5
		82.5	83.5	82.5
		84.0	83.5	82.0
		81.0	85.0	82.0
	Average	82.2	84.5	82.1
	Maximum	84.0	86.0	83.0
	Minimum	80.0	83.5	81.0

TABLE I.—RESULTS OF TESTS BY VARIOUS INVESTIGATORS ON PENETRATION OF BITUMINOUS MATERIALS (*Continued*).

	Bureau of Standards Needle No. 1	Bureau of Standards Needle No. 2	A.S.T.M. Standard Needle
SUBMITTED BY U. S. BUREAU OF STANDARDS (*Continued*)			
No. 3. Asphalt, 80 to 90 Penetration (Determination B).....	81.5 82.5 82.5 80.0 83.0 82.5 80.5 82.5 84.0 83.0	83.0 83.0 80.5 82.5 84.0 83.5 82.0 81.0 82.0 84.5	82.5 82.0 84.0 86.0 83.0 82.0 83.0 83.0 83.0 82.5
Average	82.2	82.6	83.1
Maximum	84.0	84.5	86.0
Minimum	80.0	80.5	82.0

As the present requirements permit a variation in angle of from 9 deg. 5 min. to 9 deg. 12 min., it will be noted that these needles cover a range from 33½ min. above to 29½ min. below what would be the present mean of 9 deg. 8½ min. or a total of 63 min. The report of penetration tests by the Bureau of Standards on materials of different consistencies, appearing as a part of the accompanying Table I, shows that results with these maximum and minimum needles differ from those of the A.S.T.M. standard by amounts that may be considered within the limits of ordinary experimental error.

In supplement to the foregoing, the two experimental needles have been separately examined and tested by the members of the Sectional Committee on Penetration and the results from these different laboratories have been made available. These have been compiled in a single tabulation. In all cases they corroborate the judgment of the Bureau of Standards, namely, that an angle tolerance of ± 30 min. can safely be allowed without any practical deviation from results by the present standard needle.

In view of the foregoing, and with the desire to facilitate both the manufacture and certification of A.S.T.M. penetration needles as much as possible, the Sectional Committee on Penetration recommends the revision of Section 3 of the Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5 – 21) as set forth in the Report of Committee D-4.

Opportunity is here taken to thank Messrs. Miller and Berger of the U. S. Bureau of Standards and the others who have collaborated for their interest and cooperation in this investigation.

Respectfully submitted on behalf of the sectional-committee,

LEROY M. LAW,
Chairman.

REPORT OF COMMITTEE D-5
ON
COAL AND COKE

Committee D-5 held two meetings during the year, one in June at Atlantic City, the other in March at Philadelphia. It has revised the definition of "ultimate analysis" as given in the Tentative Definitions of Terms Relating to Coal. Tests have been conducted as to methods suitable for grinding laboratory samples of coke for analysis without contamination of the sample by the grinding equipment. These tests are described in detail in Bureau of Mines *Reports of Investigations*, Serial No. 2679. Progress has been made in preparing a method of size test for anthracite. The former Sub-Committee III on Gas and Coking Coal has completed the work assigned it and has been discontinued.

PROPOSED REVISIONS OF EXISTING STANDARD METHODS OF TEST

Standard Methods of Laboratory Sampling and Analysis of Coke (D 37 - 24).[1]—Tests have been made showing that it is practicable to pulverize coke samples to pass a No. 60 sieve by means of hard-steel roll crushers without contamination of the sample. The rolls should revolve at the same speed to prevent rubbing action which might contaminate the sample through the abrasive nature of coke. Tests have also shown that considerable time is saved in pulverizing coke in ball mills if a 200-g. portion is taken instead of a 400-g. portion, as specified in the standard method. It is recommended that the following revisions in the standard methods be accepted for publication as tentative:

On page 1014 of the 1924 Book of A.S.T.M. Standards under "A Small Riffle Sampler," change "No. 10" to read "No. 20." Also change the paragraphs under "Roll Crusher" and under "Abbe Ball Mill or Hard-Steel Diamond Mortar" to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"*Hard-Steel* Roll Crusher.—For reducing the product passing a No. 4 sieve to pass a [No. 10] *No. 20* sieve."

"Abbe Ball Mill, *Hard-Steel Roll Crusher*, or Hard-Steel Diamond Mortar.—For reducing the product passing a [No. 10] *No. 20* sieve to pass a No. 60 sieve. The porcelain jars for the ball mill should be approximately 9 in. in diameter and 10 in. high. The flint pebbles should be smooth, hard, and well rounded. *The rolls of the hard-steel roll crusher should revolve at the same speed.*"

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[1] 1924 Book of A.S.T.M. Standards.

On page 1016 under "Reduction of Sample," line 9, change "No. 10 sieve" to read "No. 20 sieve;" also change "400 g." in lines 9 and 10 to read "200 g."

Change the last two sentences of the first paragraph under "Reduction of Sample" from their present form: namely,

> "Reduce the quantity of sample by quartering or riffling to about 50 g. Pass the entire 50-g. portion through a No. 60 sieve, pulverizing any coarse particles in a diamond mortar, and mix with remainder of sample, preserving the sample for analysis in a rubber-stoppered glass bottle."

to read as follows:

> "Pass all the material through a No. 60 sieve, pulverizing any coarse particles by impact in a hard-steel diamond mortar, and mix with remainder of sample. Reduce the sample through small riffle sampler to about 50 g. and transfer to a rubber-stoppered glass bottle."

Change the last paragraph under "Reduction of Sample" to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> "In case a ball mill is not available for fine grinding, quarter the 5-lb. sample passing a [No. 10] *No. 20* sieve to 200 g. and pulverize to pass a No. 60 sieve *by means of a hard-steel roll crusher in which the rolls revolve at the same speed, or* by impact in a hard-steel diamond mortar. The use of rubbing surfaces such as a disk pulverizer or a bucking board is never permissible for grinding coke."

Standard Methods of Laboratory Sampling and Analysis of Coal (D 22 - 24).[1]—A minor revision of the quantity of coal to be taken for grinding to pass a No. 60 sieve, to correspond with that proposed for coke, is desirable to make the two methods uniform in this respect. It is recommended that the following revisions in the Standard Methods of Laboratory Sampling and Analysis of Coal be accepted for publication as tentative.

On page 983 of the 1924 Book of A.S.T.M. Standards, under "Method of Sampling, (*A*) When Coal Appears Dry," line 12, change "120 g." to read "200 g."; also change "60 g." in line 6 to read "50 g." and on page 984, line 3, change "60 g." to read "about 50 g."

PROPOSED REVISIONS OF TENTATIVE STANDARDS

Tentative Definitions of Terms Relating to Coal (D 142 - 24 T).[2]—The definition of the term "ultimate analysis" has been reconsidered in view of criticisms from Committee E-8 on Nomenclature and Definitions and others, and the following revised wording is recommended:

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[1] 1924 Book of A.S.T.M. Standards.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 937 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 423.

"*Ultimate Analysis.*—In the case of coal, the quantitative determination of the elementary constituents carbon, hydrogen, and oxygen (the last by difference), in the material exclusive of incombustible residue; and the quantitative determination of sulfur and nitrogen in the material as a whole.

"NOTE 1.—Determination of phosphorus is not by definition a part of ultimate analysis of coal, but may be specified when desired.

"NOTE 2.—When the analysis is made on an undried sample, part of the hydrogen and oxygen as determined is present in the incidental or free moisture accompanying the coal. In comparing coals on the basis of their ultimate analysis, this fact is to be given due weight."

Committee D-5 has three tentative standards: namely,

1. Tentative Method of Test for Fineness of Powdered Coal (D 197 - 24 T);
2. Tentative Definitions of Terms Relating to Coke (D 121 - 24 T);
3. Tentative Definitions of Terms Relating to Coal (D 142 - 24 T).

As changes are contemplated in all of these tentative standards it is recommended that, with the proposed revision of the term "ultimate analysis," they be continued as tentative.

The result of the letter ballot vote of the committee, which consists of 44 members, on the recommendations contained in this report, is as follows:

Items	Affirmative	Negative	Not Voting
I. PROPOSED REVISIONS OF STANDARDS			
Standard Methods of Laboratory Sampling and Analysis of Coke (D 37 - 24)	36	0	8
Standard Methods of Laboratory Sampling and Analysis of Coal (D 22 - 24)	36	0	8
II. PROPOSED REVISIONS OF TENTATIVE DEFINITIONS			
Tentative Definitions of Terms Relating to Coal (D 142 - 24 T)	34	1	9

This report has been submitted to letter ballot of the committee, which consists of 44 members, of whom 36 have voted affirmatively, none negatively, and 8 have refrained from voting.

Respectfully submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

W. A. SELVIG,
Secretary.

H. C. PORTER,
Vice-Chairman.

EDITORIAL NOTE

The proposed revisions of the Standard Methods of Laboratory Sampling and Analysis of Coke; and of Laboratory Sampling and Analysis of Coal were accepted for publication as tentative and appear on pages 911 and 912.

The proposed revisions of the Tentative Definitions of Terms Relating to Coal were accepted. The tentative definitions as thus revised appear on page 752.

REPORT OF COMMITTEE D-7
ON
TIMBER

During the past year, Committee D-7 on Timber has continued its investigations looking toward the revision of the specifications for structural timbers. The Sub-Committee on Specifications for Timber prepared rather extensive revisions of the informational report submitted in 1924. The revisions included classifying and clarifying certain paragraphs and changes in the knot specifications. As the report was about to be submitted for preprinting, a report of the Central Committee on Lumber Standards was received to be acted on at a Conference called by the Secretary of Commerce, in Washington, on May 1. The recommendations made by the Central Committee on Lumber Standards, in many respects, were not in accord with the conclusions submitted by Committee D-7 a year ago and re-affirmed during the year. At the meeting of May 1 held in Washington, certain changes were made in the report of the Central Committee on Lumber Standards which bring their conclusions in accord with the recommendations of Committee D-7 and these changes were adopted by the Conference. Believing that it would be desirable to have but one standard and feeling that the members of Committee D-7 would agree to such minor changes that might be necessary in the recommendations of Committee D-7 to bring them completely in line with the report of the Central Committee on Lumber Standards, it was decided to withhold the corrected informational report of Committee D-7 until the committee could pass on these changes. The Washington Conference occurred at such a late date that it was impossible for Committee D-7 to meet to consider the suggested changes. It is planned, however, that a meeting of Committee D-7 will be held in conjunction with the annual meeting at Atlantic City and that an oral report will be made to the Society at that time, leaving the final revision of the 1924 report to be presented in printed form after the meeting.[1] It is felt that great progress has been made during the past year and that the revision of timber grades may be expected to reach a more or less definite form next year.

A strong recommendation has been made that the final revision be made in cooperation with a similar committee of the American Railway Engineering Association and other engineering bodies vitally interested in this subject.

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[1] The Report of Sub-Committee I on Specifications for Timber is appended hereto.—Ed.

Committee D-7 considered the advisability of revising the Standard Specifications for Southern Yellow-Pine Timber to be Creosoted (D 24 – 20). It has been felt for some time that the definite limitation of sap wood as given in Section 2 of these specifications should be modified. In view of the fact, however, that the specifications form an integral part of the new timber standards, it is felt that this revision should be deferred for another year.

Sub-Committee VI on Timber Preservatives has held a number of meetings during the year. A report of the sub-committee is appended hereto as information.

This report has been submitted to letter ballot of the committee, which consists of 24 members, of whom 16 have voted affirmatively, none negatively, and 8 have refrained from voting.

Respectfully submitted on behalf of the committee,

HERMANN VON SCHRENK,
Chairman.

J. A. NEWLIN,
Secretary.

REPORT OF SUB-COMMITTEE I ON SPECIFICATIONS FOR TIMBER

The Society at the present time has two standard specifications for southern yellow pine structural timbers, one for timbers to be used untreated,[1] and another for southern pine timbers to be creosoted.[2] There is also a tentative specification for Douglas fir timbers.[3] These specifications were the outcome of investigations which started early in 1905. As indicated in the 1905 report of the committee:[4] "Grading as now understood consists essentially in the specification of three factors:

(*a*) A definition of standard defects and to what extent these defects shall be admitted into various grades;

(*b*) A determination of sizes, that is, length, width and thickness;

(*c*) In certain instances, the functions to which the material is to be put are to be specified."

On this basis, the present standard and tentative specifications for structural timbers were drawn. As the result of a comprehensive study of many thousand tests the U. S. Forest Service has recently recommended a revision of these specifications for structural timbers. This revision is based upon a better understanding as to the influence of various defects on allowable stresses for timbers, under varying conditions of use. The Forest Service has suggested an approved basis for grading which consists in a closer correlation between existing defects and strength.

The sub-committee during the past two years has given serious study to these recommendations. After a detailed analysis, the general scheme employed in this basis for grading has been somewhat modified to adapt it to modern conditions of manufacture and use. Extensive revisions of the informational report published in 1924 have been made.[1] These revisions include classifying and clarifying certain paragraphs and changes in the knot specifications. As the report embodying these revisions was about to be submitted for preprinting, a report of the Central Committee on Lumber Standards was received to be acted on at a conference called by the Secretary of Commerce, in Washington, on May 1, 1925.

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[1] Standard Specifications for Yellow-Pine Bridge and Trestle Timbers (Serial Designation: D 10 – 15), 1924 Book of A.S.T.M. Standards.

[2] Standard Specifications for Southern Yellow-Pine Timber to be Creosoted (Serial Designation: D 24 – 20), 1924 Book of A.S.T.M. Standards.

[3] Tentative Specifications for Structural Douglas Fir (Serial Designation: D 23 – 20 T), *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part I, p. 704 (1920); also 1924 Book of A.S.T.M. Tentative Standards, p. 425.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. V, p. 148 (1905).

Believing that it would be desirable to have but one standard and feeling that the members of Committee D-7 would agree to such minor changes that might be necessary in the recommendations of Committee D-7 to bring them completely in line with the report of the Central Committee on Lumber Standards, it was decided to withhold the corrected informational report of Committee D-7 until the committee could pass on these changes. The Washington conference occurred at such a late date that it was impossible for Committee D-7 to meet to consider the suggested changes before the Annual Meeting.

A meeting of Committee D-7 was held in Atlantic City on June 23, 1925. The committee expressed itself as in favor of the suggested changes in the 1924 report and recommended that these new grade specifications be used in preference to the present standards and tentative standards of the Society.

The sub-committee herewith presents the results of its investigations in the form of information. The subject has been divided into:

(*a*) The basis for grading;

(*b*) Suggested grades for Douglas fir and southern pine structural material, using the new basis in the establishment of these grades;

(*c*) A suggested outline correlating these suggested grades with the functions to which the material is to be put;

(*d*) An appendix containing notes on working stresses for Douglas fir and southern pine, together with suggested values for grades complying with the basic provisions for structural material as shown in this report.

STRUCTURAL GRADES OF LUMBER AND TIMBER

Important factors which influence the strength of structural material are the size, number and location of defects, and the extent of exposure to moisture during use. These factors must all be considered in design or grading if the maximum utilization is to be obtained from the material used.

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[1] Report of Sub-Committee I on Specifications for Timber of Committee D-7 on Timber, *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 594 (1924).

Structural grades control defects by limiting their size and location in accordance with their effect upon strength. Working stresses for each species are recommended by the Forest Products Laboratory, U. S. Forest Service, which take into consideration the allowable defects, the moisture content as determined by conditions of use, and, in the case of Douglas fir and southern pine, rate of growth and percentage of summerwood.

Moisture affects the strength of structural timbers both directly and indirectly. The direct effect of loss of moisture is the stiffening and strengthening of the wood fibers. This increase in strength, however, is accompanied by checking, splitting, warping and twisting; as a consequence, some of the strength due to drying is lost. Timbers are also subject, during use, to varying conditions of moisture, from the dry condition of a heated building, to the continually wet condition of some pier and dock timbers. All of these conditions are taken into account in recommending working stresses under different conditions of use.

In dimension 4 in. and less in thickness the development of defects during seasoning does not offset the increase in strength from drying as much as in larger sizes, and in these sizes used in dry locations, higher working stresses in extreme fiber in bending can be recommended than in pieces of larger size having proportionately equivalent defects.

The principal defects which must be limited in structural grades are: knots, shakes and checks, and angle of grain.

Knots reduce the strength of a piece in somewhat direct proportion to their size, although the effect of a knot of the same proportion of the width of the face of a piece is somewhat greater in a large piece than in a smaller one, because of the greater proportional distortion of grain around a large knot than around a smaller one, and because shrinkage in seasoning causes greater proportional internal stresses around a large knot than around a smaller one. The distribution and aggregate diameter of knots is limited, as well as the maximum size of single knots.

In grades for structural uses no distinction is made between intergrown knots and encased knots or knotholes, observation at the Forest Products Laboratory in recent tests having shown that intergrown knots reduce strength fully as much as encased knots or knotholes.

Shakes reduce the area of a beam acting in resistance to shear, and the limitations placed on shake are based on this reduction. Checks are limited on the same basis as shakes, and no combination of shakes and checks is permitted which would reduce strength to a greater extent than would the allowable size of either separately.

Angle of grain, resulting either from diagonal sawing or from spiral or twisted grain in the log, is limited in accordance with the recommendations of the Forest Products Laboratory, based on the results of detailed study of the effect of cross and spiral grain upon strength, and the weakening of material by checks which invariably develop and which, without exception, follow the grain.

Wane is limited by such considerations as bearing area, nailing edge, appearance, etc., rather than by effect on strength. The percentage reduction in strength resulting from wane toward the center of a beam is about double the percentage reduction in cross-sectional area. The occurrence of maximum wane and maximum knot in the same cross-section at the center of a beam would be so rare, however, and the effect of maximum allowable wane is so small a percentage of the effect of maximum allowable knot, that the additional reduction in strength beyond the effect of the knot would be slight and it is unnecessary to give attention to combination of wane and knots.

Pitch pockets are ordinarily not defects in a structural grade. A large number, however, indicates a general lack of bond, and such a piece should be carefully inspected for shakes.

Heartwood and sapwood have been found by the Forest Products Laboratory to be of equal strength, and no requirement of heartwood need be made when strength alone is the governing factor. Heart requirement, when durability under exposure is a factor, as in bridges, trestles, docks and piers, or in damp buildings, or buildings in which conditions of high humidity prevail, may be specified in any grade, according to exposure and use.

The density of the wood substance of all species is practically the same. The dry weight is, therefore, a measure of the amount of wood substance present; and on the amount of wood substance present depends the strength of the clear wood. No pieces of exceptionally light weight are permitted in the Select grades, but very light pieces otherwise of Select grade may be accepted in the Common grades.

In Douglas fir and southern pine, the proportion of summerwood, the dark portion of the annual ring, furnishes a practical means of estimating density. Selection of these species for density, to the extent that dense material is commercially available, assures material of the highest character from the standpoint of strength, and uniformity in strength, in the clear wood.

Selection of these species for rate of growth is not as great an assurance of increased strength as selection for percentage of summerwood, but for many purposes selection for rate of growth will assure material of suitable type. Close grain, that is, not less than 6 nor more than 20 annual rings per inch, is required in the Select grades of these species.

In acceptance for density the contrast in color between summerwood and springwood should be distinct. Absence of contrast occasionally occurs in bands of growth rings which appear on the whole darker in color than the adjacent material. The summerwood merges into the springwood abnormally with a gradual change of color, leaving practically no material which has the normal appearance of springwood. Such material has been called by a number of names, including proud wood, red wood, and compression wood. It has a decided end shrinkage, is weak in tension, and is undesirable in high-class structural timbers.

Structural grades specify minimum requirements and maximum defects, all of which may be present at one time. When a particular piece which is being inspected, therefore, is slightly below the provisions of the grade in some respects but is of average density or above, the relative effect on the properties affected should be given consideration.

A large percentage of material in standard yard grades of Dimension and Timbers will meet the additional requirements of structural grades for Joist and Plank, and Posts and Timbers, and material to meet these requirements can easily be selected from local stocks: Select from Select Common and Merchantable grades, and Common from No. 1 Common grade. Beams and Stringers vary materially in size and are not stocked extensively. These are essentially special order grades.

The provisions of the Joist and Plank grades are such that material graded by them may be used on edge, as joists or rafters, or flat, as scaffold plank or factory flooring. Joist and Plank grades apply to material not thicker than 4 in. Material thicker than 4 in., for use in bending, should be graded by Beam and Stringer grades.

Material to be used for such purposes as caps, bridge ties, etc., where strength in bending is a factor, should be specified in Beam and Stringer grades although of shape more commonly considered as of Timber grades, as the method of measuring knots in Post and Timber structural grades makes it impracticable to assign bending stresses to these grades.

The following provisions were accepted at the General Lumber Conference, Washington, D. C., May 1, 1925, as the basis for the preparation of grading rules for structural material where working stresses are required. Material for any of the three "Uses" can be specified in any of the three "Grades," making nine "Use-Grades" available.

GRADES AND CLASSIFICATIONS

Grades: DENSE SELECT;
SELECT;
COMMON.

Uses: JOIST AND PLANK;
Joists, Rafters, Scaffold Plank, Factory Flooring, etc.

BEAMS AND STRINGERS;
Beams, Girders, Stringers, etc.

POSTS AND TIMBERS.
Posts, Caps, Sills, Timbers, etc.

General Provisions

All grades shall contain only sound wood, unless decay is specifically permitted.

The measurement of a knot shall be made on the section of the knot appearing on the surface under consideration.

In Post and Timber grades, and on the wide faces of Joist and Plank, the measurement of a knot shall be made on the mean or average diameter.

On the narrow faces of Joist and Plank, and Beams and Stringers, the size of a knot shall be taken as its width between lines parallel to the edges of the timber.

On the wide or vertical faces of Beams and Stringers, the smallest diameter of a knot shall be taken as its size.

Knots on the edges of wide faces of Beams and Stringers are limited to the same size as on the adjacent narrow faces.

Knots on the narrow faces and edges of wide faces of Joist and Plank, and Beams and Stringers, may increase proportionately from the size allowed in the middle third to twice that size at the ends of the piece.

The size of knots on the wide faces of Joist and Plank, and Beams and Stringers, may increase proportionately from the size allowed at the edge to that allowed at the center line.

Cluster knots and knots in groups are not permitted.

Knot holes and holes from other causes than knots shall be limited as provided for knots.

Shake shall be measured on the end of a piece, and its size shall be taken as the shortest distance between lines enclosing the shake and parallel to the wide faces of the piece. Checks and splits shall be limited as provided for shakes. No checks or combinations of checks with shakes which would reduce the strength to a greater extent than the allowable shake shall be permitted.

No combination of wane and knots is permitted which would reduce the strength more than the maximum allowable knot.

No pieces of exceptionally light weight shall be permitted in any grade, except that very light pieces otherwise of Select grade may be accepted in Common grade.

Special Provisions

No heartwood requirements are provided in these grades. Heartwood requirements when ordered should be specified in terms of heartwood required on the girth or on each face, side or edge. Girth shall be measured at the point where the greatest amount of sapwood occurs.

Wane is permitted in all grades, but square edges may be specified when appearance or use require.

Methods of measurement of Douglas fir and southern pine for rate of growth and density are given for use with the Dense Select and Select grades.

Selection for Rate of Growth, Douglas Fir and Southern Pine

Douglas fir or southern pine selected for close grain shall average on either one end or the other not less than 6 nor more than 20 annual rings per inch, measured over a 3-in. portion of a radial line, representative of the average growth on the cross-section, located as described below.

When such radial line is not representative, it shall be shifted sufficiently to present a fair average, but the distance from the pith of the 3-in. portion of the line shall not be changed.

In case of disagreement two radial lines shall be chosen, and the number of rings shall be the average of these lines.

Douglas Fir.—In side-cut pieces the line shall be at a right angle to the annual rings and the center of the 3-in. portion of the line shall be at the center of the end of the piece.

In boxed-heart pieces the line shall run from the pith to the corner farthest from the pith. When the least dimension is 6 in. or less, the 3-in. portion of the line shall begin at a distance of 1 in. from the pith. When the least dimension is more than 6 in., the 3-in. portion of the line shall begin at a distance from the pith equal to 2 in. less than one-half the least dimension of the piece.

If a 3-in. portion of the radial line cannot be obtained, the measurement shall be made over as much of the 3-in. portion as is available.

Southern Pine.—In boxed-heart pieces the rate of growth shall be counted over the third, fourth and fifth inches from the pith along the radial line.

In cases where timbers do not contain the pith, and it is impossible to locate it with any degree of accuracy, the same inspection shall be made over 3 in. on an approximate radial line beginning at the edge nearest the pith in timbers over 3 in. in thickness and on the second inch nearest to the pith in timbers 3 in. or less in thickness.

In material containing the pith but not a 5-in. radial line, which is less than 2 by 8 in. in section or less than 8 in. in width, that does not show over 16 sq. in. on the cross-section, the inspection shall apply to the second inch from the pith. In larger material that does not show a 5-in. radial line, the inspection shall apply to the 3 in. farthest from the pith.

Selection for Density, Douglas Fir and Southern Pine

Douglas fir or southern pine selected for density shall average on either one end or the other not less than 6 annual rings per inch and in addition one-third, or more, summerwood, over the same portion of a radial line as provided for selection for rate of growth. Coarse-grained material excluded by this rule shall be accepted as dense if averaging one-half, or more, summerwood.

The contrast in color between summerwood and springwood shall be sharp.

In case of disagreement two radial lines shall be chosen, and the summerwood and number of rings shall be the average of these lines.

BASIC GRADES AS APPLIED TO DOUGLAS FIR AND SOUTHERN PINE

I. DENSE SELECT GRADE TIMBERS

1. Douglas fir and southern pine timbers conforming to the following requirements for density:

Douglas fir or southern pine selected for density shall average on either one end or the other not less than 6 annual rings per inch and in addition one-third, or more, summerwood, over the same portion of a radial line as provided for selection for rate of growth. Coarse-grained material excluded by this rule shall be accepted as dense if averaging one-half, or more, summerwood.

The contrast in color between summerwood and springwood shall be sharp.

When such radial line is not representative, it shall be shifted sufficiently to present a fair average, but the distance from the pith of the 3-in. portion of the line shall not be changed.

In case of disagreement two radial lines shall be chosen, and the summerwood and number of rings shall be the average of these lines.

Douglas Fir.—In side-cut pieces the line shall be at a right angle to the annual rings and the center of the 3-in. portion of the line shall be at the center of the end of the piece.

In boxed-heart pieces the line shall run from the pith to the corner farthest from the pith. When the least dimension is 6 in. or less, the 3-in. portion of the line shall begin at a distance of 1 in. from the pith. When the least dimension is more than 6 in., the 3-in. portion of the line shall begin at a distance from the pith equal to 2 in. less than one-half the least dimension of the piece.

If a 3-in. portion of the radial line cannot be obtained, the measurement shall be made over as much of the 3-in. portion as is available.

Southern Pine.—In boxed-heart pieces the rate of growth shall be counted over the third, fourth and fifth inches from the pith along the radial line.

In cases where timbers do not contain the pith, and it is impossible to locate it with any degree of accuracy, the same inspection shall be made over 3 in. on an approximate radial line beginning at the edge nearest the pith in timbers over 3 in. in thickness and on the second inch nearest to the pith in timbers 3 in. or less in thickness.

In material containing the pith but not a 5-in. radial line, which is less than 2 by 8 in. in section or less than 8 in. in width, that does not show over 16 sq. in. on the cross-section, the inspection shall apply to the second inch from the pith. In larger material that does not show a 5-in. radial line, the inspection shall apply to the 3 in. farthest from the pith.

BEAMS AND STRINGERS

Basic Provisions

2. *Knots.* (*a*) *General.*—The sum of the diameters of all knots within the center half of the length of a beam shall not exceed the width of the face on which they occur.

(*b*) *Maximum Size.*—The maximum size of knots shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM SIZE OF KNOTS, IN. Narrow Faces and Edges of Wide Faces, Middle Third of Length	Center Line Wide Faces
5	1¼	—
6	1½	1½
8	1¾	2
10	2	2½
12	2⅛	3
14	2¼	3¼
16	2⅜	3⅜
18	—	3⅝
20	—	3⅞
22	—	4
24	—	4¼

NOTE.—Knots on narrow faces and edges of wide faces may increase proportionately from the size allowed at the edge to that allowed at the center line.

Increase in size of knots on wide face is proportional to distance of center line of knot from edge of wide face, to a maximum at the center line of wide face.

3. *Shakes.*—The maximum width of shakes shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM WIDTH OF SHAKES, IN. Green	Seasoned
6	1½	2
8	2	2⅝
10	2½	3¼
12	3	4
14	3½	4⅝
16	4	5¼
18	4½	6
20	5	6⅝
22	5½	7¼
24	6	8

4. *Angle of Grain.*—The angle of grain in the center half of the length shall not exceed 1 in 15.

5. *Wane.*—Wane shall not exceed one-eighth of the width of the face on which it occurs.

Special Provisions

6. *Heartwood.*—When resistance to decay without preservative treatment is a factor, 85 per cent heart on each of the four sides should be specified. See A.S.T.M. Tentative Specifications for Douglas Fir (D 23 – 20 T)[1] and A.S.T.M. Standard Specifications for Yellow-Pine Bridge and Trestle Timbers (D 10 – 15).[2]

7. *Sapwood.*—When preservative treatment is to be applied, there shall be no restriction as to sapwood, that is, a large amount of sapwood will be preferred.

8. *Square Edge.*—When appearance or bearing are factors, square edge timbers should be specified.

POSTS AND TIMBERS

Basic Provisions

9. *Knots, Maximum Size.*—The maximum size of knots shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM SIZE OF KNOTS, IN.
6	1½
8	2
10	2½
12	3
14	3¼
16	3⅜
18	3⅝
20	3⅞
22	4
24	4¼

10. *Shakes.*—The maximum width of shakes shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM WIDTH OF SHAKES, IN.	
	Green	Seasoned
5	2	2½
6	2⅜	3
8	3⅛	4
10	4	5
12	4¾	6
14	5½	7
16	6⅜	8
18	7⅛	9
20	8	10
22	8¾	11
24	9½	12

11. *Angle of Grain.*—The angle of grain shall not exceed 1 in 10.

12. *Wane.*—Wane shall not exceed one-eighth of the width of the face on which it occurs.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part I, p. 704 (1920); also 1924 Book of A.S.T.M. Tentative Standards, p. 425. These specifications will hereafter be referred to in the abbreviated form: A.S.T.M. Specifications D 23 – 20 T.

[2] 1924 Book of A.S.T.M. Standards. These specifications will hereafter be referred to in the abbreviated form: A.S.T.M. Specifications D 10 – 15.

Special Provisions

13. *Heartwood.*—When resistance to decay without preservative treatment is a factor, 75 per cent heart on each of the four sides should be specified. See A.S.T.M. Specifications D 23 - 20 T for Douglas fir and A.S.T.M. Specifications D 10 - 15 for southern pine.

14. *Sapwood.*—When preservative treatment is to be applied, there shall be no restriction as to sapwood, that is, a large amount of sapwood will be preferred.

15. *Square Edge.*—When appearance is a factor, square-edge timbers should be specified.

II. SELECT GRADE TIMBERS

16. Douglas fir or southern pine of close grain conforming to the following requirements:

Douglas fir or southern pine selected for close grain shall average on either one end or the other not less than 6 nor more than 20 annual rings per inch, measured over a 3-in. portion of a radial line, representative of the average growth on the cross-section, located as described below.

When such radial line is not representative, it shall be shifted sufficiently to present a fair average, but the distance from the pith of the 3-in. portion of the line shall not be changed.

In case of disagreement two radial lines shall be chosen, and the number of rings shall be the average of these lines.

Douglas Fir.—In side-cut pieces the line shall be at a right angle to the annual rings and the center of the 3-in. portion of the line shall be at the center of the end of the piece.

In boxed-heart pieces the line shall run from the pith to the corner farthest from the pith. When the least dimension is 6 in. or less, the 3-in. portion of the line shall begin at a distance of 1 in. from the pith. When the least dimension is more than 6 in., the 3-in. portion of the line shall begin at a distance from the pith equal to 2 in. less than one-half the least dimension of the piece.

If a 3-in. portion of the radial line cannot be obtained, the measurement shall be made over as much of the 3-in. portion as is available.

Southern Pine.—In boxed-heart pieces the rate of growth shall be counted over the third, fourth and fifth inches from the pith along the radial line.

In cases where timbers do not contain the pith, and it is impossible to locate it with any degree of accuracy, the same inspection shall be made over 3 in. on an approximate radial line beginning at the edge nearest the pith in timbers over 3 in. in thickness and on the second inch nearest to the pith in timbers 3 in. or less in thickness.

In material containing the pith but not a 5-in. radial line, which is less than 2 by 8 in. in section or less than 8 in. in width, that does not show over 16 sq. in. on the cross-section, the inspection shall apply to the second inch from the pith. In larger material that does not show a 5-in. radial line, the inspection shall apply to the 3 in. farthest from the pith.

BEAMS AND STRINGERS

Basic Provisions

17. *Knots.* (*a*) *General.*—The sum of the diameters of all knots within the center half of the length of a beam shall not exceed the width of the face on which they occur.

(*b*) *Maximum Size.*—The maximum size of knots shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM SIZE OF KNOTS, IN. Narrow Faces and Edges of Wide Faces, Middle Third of Length	MAXIMUM SIZE OF KNOTS, IN. Center Line Wide Faces
5	1¼	—
6	1½	1½
8	1¾	2
10	2	2½
12	2⅛	3
14	2¼	3¼
16	2⅜	3⅜
18	—	3⅝
20	—	3⅞
22	—	4
24	—	4¼

NOTE.—Knots on narrow faces and edges of wide faces may increase proportionately from the size allowed at the edge to that allowed at the center line.

Increase in size of knot on wide face is proportional to distance of center line of knot from edge of wide face, to a maximum at the center line of wide face.

18. *Shakes.*—The maximum width of shakes shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM WIDTH OF SHAKES, IN. Green	MAXIMUM WIDTH OF SHAKES, IN. Seasoned
6	1½	2
8	2	2⅝
10	2½	3¼
12	3	4
14	3½	4⅝
16	4	5¼
18	4½	6
20	5	6⅝
22	5½	7¼
24	6	8

19. *Angle of Grain.*—The angle of grain in the center half of the length shall not exceed 1 in 15.

20. *Wane.*—Wane shall not exceed one-eighth of the width of the face on which it occurs.

Special Provisions

21. *Heartwood.*—When resistance to decay without preservative treatment is a factor, 85 per cent heart on each of the four sides should be specified. See A.S.T.M. Specifications D 23 - 20 T for Douglas fir and A.S.T.M. Specifications D 10 - 15 for southern pine.

22. *Sapwood.*—When preservative treatment is to be applied, there shall be no restriction as to sapwood, that is, a large amount of sapwood will be preferred.

23. *Square Edge.*—When appearance or bearing are factors, square edge timbers should be specified.

POSTS AND TIMBERS

Basic Provisions

24. *Knots, Maximum Size.*—The maximum size of knots shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM SIZE OF KNOTS, IN.
6	1½
8	2
10	2½
12	3
14	3¼
16	3⅜
18	3⅝
20	3⅞
22	4
24	4¼

25. *Shakes.*—The maximum width of shakes shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM WIDTH OF SHAKES, IN.	
	Green	Seasoned
5	2	2½
6	2⅜	3
8	3⅛	4
10	4	5
12	4¾	6
14	5½	7
16	6⅜	8
18	7⅛	9
20	8	10
22	8¾	11
24	9½	12

26. *Angle of Grain.*—The angle of grain shall not exceed 1 in 10.

27. *Wane.*—Wane shall not exceed one-eighth of the width of the face on which it occurs.

Special Provisions

28. *Heartwood.*—When resistance to decay without preservative treatment is a factor, 75 per cent heart on each of the four sides should be specified. See A.S.T.M. Specifications D 23 - 20 T for Douglas fir and A. S. T. M. Specifications D 10 - 15 for southern pine.

29. *Sapwood.*—When preservative treatment is to be applied, there shall be no restriction as to sapwood, that is, a large amount or sapwood will be preferred.

30. *Square Edge.*—When appearance is a factor, square-edge timbers should be specified.

III. COMMON GRADE TIMBERS

31. Douglas fir or southern pine timbers.

BEAMS AND STRINGERS

Basic Provisions

32. *Knots.* (*a*) *General.*—The sum of the diameters of all knots within the center half of the length of a beam shall not exceed $1\frac{1}{2}$ times the width of the face on which they occur.

(*b*) *Maximum Size.*—The maximum size of knots shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM SIZE OF KNOTS, IN.	
	Narrow Faces and Edges of Wide Faces, Middle Third of Length	Center Line Wide Faces
5	2	—
6	$2\frac{3}{8}$	$2\frac{3}{8}$
8	$2\frac{3}{4}$	$3\frac{1}{8}$
10	$3\frac{1}{8}$	4
12	$3\frac{3}{8}$	$4\frac{3}{4}$
14	$3\frac{5}{8}$	$5\frac{1}{8}$
16	$3\frac{7}{8}$	$5\frac{1}{2}$
18	—	$5\frac{7}{8}$
20	—	$6\frac{1}{8}$
22	—	$6\frac{1}{2}$
24	—	$6\frac{3}{4}$

NOTE.—Knots on narrow faces and edges of wide faces may increase proportionately from the size allowed at the edge to that allowed at the center line.

Increase in size of knot on wide face is proportional to distance of center line of knot from edge of wide face, to a maximum at the center line of wide face.

33. *Shakes.*—The maximum width of shakes shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM WIDTH OF SHAKES, IN.	
	Green	Seasoned
5	2	$2\frac{1}{8}$
6	$2\frac{3}{8}$	$2\frac{5}{8}$
8	$3\frac{1}{8}$	$3\frac{1}{2}$
10	4	$4\frac{3}{8}$
12	5	$5\frac{1}{4}$
14	$5\frac{1}{2}$	$6\frac{1}{8}$
16	$6\frac{3}{8}$	7
18	$7\frac{1}{8}$	8
20	8	$8\frac{7}{8}$
22	$8\frac{3}{4}$	$9\frac{3}{4}$
24	$9\frac{1}{2}$	$10\frac{5}{8}$

34. *Angle of Grain.*—The angle of grain in the center half of the length shall not exceed 1 in 10.

35. *Wane.*—Wane shall not exceed one-fourth of the width of the face on which it occurs.

Special Provisions

36. *Heartwood.*—When resistance to decay without preservative treatment is a factor, 85 per cent heart on each of the four sides should be specified. See A.S.T.M. Specifications D 23 - 20 T for Douglas fir and A.S.T.M. Specifications D 10 - 15 for southern pine.

37. *Sapwood.*—When preservative treatment is to be applied, there shall be no restriction as to sapwood, that is, a large amount of sapwood will be preferred.

38. *Square Edge.*—When appearance or bearing are factors, square-edge timbers should be specified.

POSTS AND TIMBERS

Basic Provisions

39. *Knots, Maximum Size.*—The maximum size of knots shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM SIZE OF KNOT, IN.
6	$2\frac{3}{8}$
8	$3\frac{1}{8}$
10	4
12	$4\frac{3}{4}$
14	$5\frac{1}{8}$
16	$5\frac{1}{2}$
18	$5\frac{7}{8}$
20	$6\frac{1}{8}$
22	$6\frac{1}{2}$
24	$6\frac{3}{4}$

40. *Shakes.*—The maximum width of shakes shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM WIDTH OF SHAKES, IN.	
	Green	Seasoned
6	3	$3\frac{1}{2}$
8	4	$4\frac{3}{4}$
10	5	6
12	6	$7\frac{1}{8}$
14	7	$8\frac{3}{8}$
16	8	$9\frac{1}{2}$
18	9	$10\frac{3}{4}$
20	10	12
22	11	$13\frac{1}{8}$
24	12	$14\frac{3}{8}$

41. *Angle of Grain.*—The angle of grain shall not exceed 1 in 8.

42. *Wane.*—Wane shall not exceed one-fourth of the width of the face on which it occurs.

Special Provisions

43. *Heartwood.*—When resistance to decay without preservative treatment is a factor, 75 per cent heart on each of the four sides should be specified. See

A.S.T.M. Specifications D 23 – 20 T for Douglas fir and A.S.T.M. Specifications D 10 – 15 for southern pine.

44. *Sapwood.*—When preservative treatment is to be applied, there shall be no restriction as to sapwood, that is, a large amount of sapwood will be preferred.

45. *Square Edge.*—When appearance is a factor, square-edge timbers should be specified.

IV. DENSE SELECT GRADE JOIST AND PLANK

46. Douglas fir or southern pine dimension conforming to the following requirements for density:

Douglas fir or southern pine selected for density shall average on either one end or the other not less than 6 annual rings per inch and in addition one-third, or more, summerwood, over the same portion of a radial line as provided for selection for rate of growth. Coarse-grained material excluded by this rule shall be accepted as dense if averaging one-half, or more, summerwood.

The contrast in color between summerwood and springwood shall be sharp.

When such radial line is not representative, it shall be shifted sufficiently to present a fair average, but the distance from the pith of the 3-in. portion of the line shall not be changed.

In case of disagreement, two radial lines shall be chosen, and the summerwood and number of rings shall be the average of these lines.

Pieces thicker than 4 in. shall be graded in accordance with structural grades for beams and stringers in timbers.

Douglas Fir.—In side-cut pieces the line shall be at a right angle to the annual rings and the center of the 3-in. portion of the line shall be at the center of the end of the piece.

In boxed-heart pieces the line shall run from the pith to the corner farthest from the pith. When the least dimension is 6 in. or less, the 3-in. portion of the line shall begin at a distance of 1 in. from the pith.

If a 3-in. portion of the radial line cannot be obtained, the measurement shall be made over as much of the 3-in. portion as is available.

Southern Pine.—In boxed-heart pieces the rate of growth shall be counted over the third, fourth and fifth inches from the pith along the radial line.

In cases where timbers do not contain the pith, and it is impossible to locate it with any degree of accuracy, the same inspection shall be made over 3 in. on an approximate radial line beginning at the edge nearest the pith in timbers over 3 in. in thickness and on the second inch nearest to the pith in timbers 3 in. or less in thickness.

In material containing the pith but not a 5-in. radial line, which is less than 2 by 8 in. in section or less than 8 in. in width, that does not show over 16 sq. in. on the cross-section, the inspection shall apply to the second inch from the pith. In larger material that does not show a 5-in. radial line, the inspection shall apply to the 3 in. farthest from the pith.

Basic Provisions

47. *Knots.* (*a*) *General.*—The sum of the diameters of all knots within the center half of the length of a beam shall not exceed 1½ times the width of the face on which they occur. Knots on wide faces are measured as of their average diameter.

(*b*) ***Maximum Size on Wide Faces.***—The maximum size of knots on wide faces shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM SIZE OF KNOTS, IN. On or Near Edge, Middle Third of Length	MAXIMUM SIZE OF KNOTS, IN. Center Line of Face
4	¾	1¼
6	1	2
8	1⅜	2⅝
10	1¾	3¼
12	2⅛	4
14	2⅜	4¼
16	2½	4⅝

(*c*) ***Maximum Size on Narrow Faces, Boxed-Heart Pieces.***—The maximum size of knots on narrow faces of boxed-heart pieces, middle third of length, shall be as follows:

WIDTH OF FACE, IN.	SIZE OF KNOTS, IN.
2	⅝
3	1
4	1¼

NOTE.—Knots on narrow faces and edges of wide faces may increase proportionately from the size allowed at the edge to that allowed at the center line.

Increase in size of knot on wide face is proportional to distance of center line of knot from edge of wide face, to a maximum at the center line of wide face.

48. *Shakes.*—The maximum width of shakes shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM WIDTH OF SHAKES, IN. Green	MAXIMUM WIDTH OF SHAKES, IN. Seasoned
2	½	⅝
3	¾	1
4	1	1¼

49. *Angle of Grain.*—The angle of grain in the center half of the length shall not exceed 1 in 12.

50. *Wane.*—Wane shall not exceed one-eighth of the width of the face on which it occurs.

Special Provisions

51. *Heartwood.*—When resistance to decay without preservative treatment is a factor, 85 per cent heart on each of the four sides should be specified. See A.S.T.M. Specifications D 23 – 20 T for Douglas fir and A.S.T.M. Specifications D 10 – 15 for southern pine.

52. *Sapwood.*—When preservative treatment is to be applied, there shall be no restriction as to sapwood, that is, a large amount of sapwood will be preferred.

53. *Square Edge.*—When appearance or bearing are factors, square-edge pieces should be specified.

V. SELECT GRADE JOIST AND PLANK

54. Douglas fir or southern pine of close grain conforming to the following requirements:

Douglas fir or southern pine selected for close grain shall average on either one end or the other not less than 6 nor more than 20 annual rings per inch, measured over a 3-in. portion of a radial line, representative of the average growth on the cross-section, located as described below.

When such radial line is not representative, it shall be shifted sufficiently to present a fair average, but the distance from the pith of the 3-in. portion of the line shall not be changed.

In case of disagreement two radial lines shall be chosen, and the number of rings shall be the average of these lines.

Pieces thicker than 4 in. shall be graded in accordance with structural grades for beams and stringers in timbers.

Douglas Fir.—In side-cut pieces the line shall be at a right angle to the annual rings and the center of the 3-in. portion of the line shall be at the center of the end of the piece.

In boxed-heart pieces the line shall run from the pith to the corner farthest from the pith. When the least dimension is 6 in. or less, the 3-in. portion of the line shall begin at a distance of 1 in. from the pith.

If a 3-in. portion of the radial line cannot be obtained, the measurement shall be made over as much of the 3-in. portion as is available.

Southern Pine.—In boxed-heart pieces the rate of growth shall be counted over the third, fourth and fifth inches from the pith along the radial line.

In cases where timbers do not contain the pith, and it is impossible to locate it with any degree of accuracy, the same inspection shall be made over 3 in. on an approximate radial line beginning at the edge nearest the pith in timbers over 3 in. in thickness and on the second inch nearest to the pith in timbers 3 in. or less in thickness.

In material containing the pith but not a 5-in. radial line, which is less than 2 by 8 in. in section or less than 8 in. in width, that does not show over 16 sq. in. on the cross-section, the inspection shall apply to the second inch from the pith. In larger material that does not show a 5-in. radial line, the inspection shall apply to the 3 in. farthest from the pith.

Basic Provisions

55. *Knots.* (*a*) *General.*—The sum of the diameters of all knots within the center half of the length of a beam shall not exceed 1½ times the width of the face on which they occur. Knots on wide faces are measured as of their average diameter.

(*b*) *Maximum Size on Wide Faces.*—The maximum size of knots on wide faces shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM SIZE OF KNOTS, IN.	
	On or Near Edge, Middle Third of Length	Center Line of Face
4	$\frac{3}{4}$	$1\frac{1}{4}$
6	1	2
8	$1\frac{3}{8}$	$2\frac{5}{8}$
10	$1\frac{3}{4}$	$3\frac{1}{4}$
12	$2\frac{1}{8}$	4
14	$2\frac{3}{8}$	$4\frac{1}{4}$
16	$2\frac{1}{2}$	$4\frac{5}{8}$

(*c*) *Maximum Size on Narrow Faces, Boxed-Heart Pieces.*—The maximum size of knots on narrow faces of boxed-heart pieces, middle third of length, shall be as follows:

WIDTH OF FACE, IN.	SIZE OF KNOTS, IN.
2	$\frac{5}{8}$
3	1
4	$1\frac{1}{4}$

NOTE.—Knots on narrow faces and edges of wide faces may increase proportionately from the size allowed at the edge to that allowed at the center line.

Increase in size of knot on wide face is proportional to distance of center line of knot from edge of wide face, to a maximum at the center line of wide face.

56. *Shakes.*—The maximum width of shakes shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM WIDTH OF SHAKES, IN.	
	Green	Seasoned
2	$\frac{1}{2}$	$\frac{5}{8}$
3	$\frac{3}{4}$	1
4	1	$1\frac{1}{4}$

57. *Angle of Grain.*—The angle of grain in the center half of the length shall not exceed 1 in 12.

58. *Wane.*—Wane shall not exceed one-eighth of the width of the face on which it occurs.

Special Provisions

59. *Heartwood.*—When resistance to decay without preservative treatment is a factor, 85 per cent heart on each of the four sides should be specified. See A.S.T.M. Specifications D 23 – 20 T for Douglas fir and A.S.T.M. Specifications D 10 – 15 for southern pine.

60. *Sapwood.*—When preservative treatment is to be applied, there shall be no restriction as to sapwood, that is, a large amount of sapwood will be preferred.

61. *Square Edge.*—When appearance or bearing are factors, square-edge pieces should be specified.

VI. COMMON GRADE JOIST AND PLANK

62. Douglas fir or southern pine. Pieces thicker than 4 in. shall be graded in accordance with structural grades for beams and stringers in timbers.

Basic Provisons

63. *Knots.* (*a*) *General.*—The sum of the diameters of all knots within the center half of the length of a beam shall not exceed 2 times the width of the face on which they occur. Knots on wide faces are measured as of their average diameter.

(*b*) *Maximum Size on Wide Faces.*—The maximum size of knots on wide faces shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM SIZE OF KNOTS, IN	
	On or Near Edge, Middle Third of Length	Center Line of Face
4	1	$1\frac{3}{4}$
6	$1\frac{1}{2}$	$2\frac{1}{2}$
8	2	$3\frac{3}{8}$
10	$2\frac{1}{2}$	$4\frac{1}{4}$
12	3	$5\frac{1}{8}$
14	$3\frac{1}{4}$	$5\frac{5}{8}$
16	$3\frac{3}{8}$	6

(*c*) *Maximum Size on Narrow Faces, Boxed-Heart Pieces.*—The maximum size of knots on narrow faces of boxed-heart pieces, middle third of length, shall be as follows:

WIDTH OF FACE, IN.	SIZE OF KNOTS. IN.
2	$\frac{7}{8}$
3	$1\frac{1}{4}$
4	$1\frac{3}{4}$

NOTE.—Knots on narrow faces and edges of wide faces may increase proportionately from the size allowed at the edge to that allowed at the center line.

Increase in size of knot on wide face is proportional to distance of center line of knot from edge of wide face, to a maximum at the center line of wide face.

64. *Shakes.*—The maximum width of shakes shall be as follows:

WIDTH OF FACE, IN.	MAXIMUM WIDTH OF SHAKES, IN.	
	Green	Seasoned
2	$\frac{3}{4}$	$\frac{7}{8}$
3	$1\frac{1}{8}$	$1\frac{1}{4}$
4	$1\frac{1}{2}$	$1\frac{3}{4}$

65. *Angle of Grain.*—The angle of grain in the center half of the length shall not exceed 1 in 10.

66. *Wane.*—Wane shall not exceed one-fourth of the width of the face on which it occurs.

Special Provisions

67. *Heartwood.*—When resistance to decay without preservative treatment is a factor, 85 per cent heart on each of the four sides should be specified. See A.S.T.M. Specifications D 23 - 20 T for Douglas fir and A.S.T.M. Specifications D 10 - 15 for southern pine.

68. *Sapwood.*—When preservative treatment is to be applied, there shall be no restriction as to sapwood, that is, a large amount of sapwood will be preferred.

69. *Square Edge.*—When appearance or bearing are factors, square edges should be specified.

USE SPECIFICATIONS FOR TIMBER AND LUMBER (LOAD BEARING)

The quality of timbers or lumber best suited for load-bearing uses in a given structure varies with the kind and amount of load and the liability of deterioration by decay. Safety in design, resistance to decay and economy in cost may be regulated by a study of the use of the structure and its degree of exposure.

Grades of timbers may be specified in any case to meet any of three use viewpoints:

1. Load carrying capacity, which will mean the use of Dense Select, Select, or Common timbers, depending upon the loads to be carried and space limitations.

2. Decay resistance, which places a heartwood requirement on any of these three grades of timbers if they are to be used in damp or exposed locations without preservative treatment, or a sapwood requirement if they are to be treated.

3. A combination of 1 and 2.

Where details of design will permit, it may be possible to substitute grades lower than specified in the Use Specifications which follow, allowing the increased size of the member of lower strength quality to fulfill the requirements of strength. This does not alter the need for heartwood or sapwood requirements. While there is no difference in strength between heartwood and sapwood, each has its function in resisting decay or aiding preservative treatment.

In a similar manner, treated timbers of low strength quality often may be used in locations where heartwood timbers of higher quality and cost would be used.

When appearance is a factor, square edge timbers should be specified.

TYPES OF STRUCTURES

Load-bearing structures may be classified under the following general divisions:

A. Buildings;
B. Bridges and Trestles;
C. Docks, Piers and Wharves;
D. Platforms;
E. Gates, Bins, Bulkheads and Sheet Piling;
F. Cars;
G. Barges;
H. Transmission Lines;
I. Track;
J. False Work and Temporary Construction;
K. Mine Supports.

In addition to these general divisions, there are many auxiliary uses for timbers and lumber where isolated members form a part of the general structure. The importance of these members may be great and may warrant a Use Specification in many cases; but in general these cases will be covered by the specifications for members forming parts of the general divisions shown above, where such members are used under similar stress and exposure conditions.

There are given below two suggested Use Specifications covering the first two classifications of the above types of structures, namely, buildings (mill construction type) and bridges and trestles. The committee is now engaged in developing Use Specifications for other classes of structures listed above.

USE SPECIFICATIONS (*A*): BUILDINGS

(MILL CONSTRUCTION TYPE)

I. HEAVY LOADING

(LIVE FLOOR LOAD OVER 200 LB. PER SQ. FT.)

Girders.—Dense Select Grade Douglas fir or southern pine.
Beams.—Dense Select Grade Douglas fir or southern pine.
Posts.—Select Grade Douglas fir or southern pine.
Sub-Floor.—Select Grade Douglas fir or southern pine.
Roof Plank.—Common Grade Douglas fir or southern pine.

II. MEDIUM LOADING

(LIVE FLOOR LOAD 200 LB. PER SQ. FT. AND UNDER)

Girders.—Select Grade Douglas fir or southern pine.
Beams.—Select Grade Douglas fir or southern pine.
Posts.—Common Grade Douglas fir or southern pine.

Sub-Floor.—Common Grade Douglas fir or southern pine.

Roof Plank.—Common Grade Douglas fir or southern pine.

NOTE.—In cases of extreme moisture or where manufacturing processes are likely to favor decay, a heartwood requirement should be included unless a recognized preservative treatment is applied to the grades named, when a sapwood requirement should be included. For details, see reference in various grades.

USE SPECIFICATIONS (*B*): BRIDGES AND TRESTLES

I. HEAVY LOADING, RAILWAY

(COOPER'S CLASS E-60 AND ABOVE)

Stringers.—Dense Select Grade Douglas fir or southern pine.

Caps.—Select Grade Douglas fir or southern pine.

Posts.—Select Grade Douglas fir or southern pine.

Sills.—Common Grade Douglas fir or southern pine.

Braces.—Select Grade Douglas fir or southern pine. Heartwood requirement or preservative treatment.

Longitudinal Struts or Girts—Select Grade Douglas fir or southern pine. Heartwood requirement or preservative treatment.

Bridge Ties.—Select Grade Douglas fir or southern pine. Heartwood requirement or preservative treatment.

II. MEDIUM LOADING, RAILWAY OR HIGHWAY

(BELOW COOPER'S CLASS E-60)

Stringers.—Select Grade Douglas fir or southern pine. Heartwood requirement or preservative treatment.

Caps.—Select Grade Douglas fir or southern pine.

Posts.—Common Grade Douglas fir or southern pine.

Sills.—Common Grade Douglas fir or southern pine.

Braces.—Common Grade Douglas fir or southern pine. Heartwood requirement or preservative treatment.

Longitudinal Struts or Girts.—Common Grade Douglas fir or southern pine. Heartwood requirement or preservative treatment.

Bridge Ties.—Common Grade Douglas fir or southern pine. Heartwood requirement or preservative treatment.

Planking.—Select Grade Douglas fir or southern pine.

This report is presented as information in order to bring out discussion of the general scheme of classification of grades and types of structure as outlined.

Respectfully submitted on behalf of the sub-committee,

C. E. PAUL,
Chairman.

APPENDIX[1]

WORKING STRESSES FOR DOUGLAS FIR AND SOUTHERN PINE

NOTES ON TABLE OF WORKING STRESSES

The purpose of structural grades is to offer means for selecting structural material for strength, and for uniformity in strength, and appropriate working stresses are essential to their use.

The working stresses in the accompanying table are recommended by the Forest Products Laboratory, U. S. Forest Service, for structural grades complying with Basic Provisions for Structural Material of American Lumber Standards.

Working stresses depend on the strength of clear wood and, in some properties, on conditions of exposure during use, on size of piece, and on grade as fixed by limitation on size and location of knots, extent of shake and checks, and extent of cross grain. In Douglas fir and southern pine, working stresses in some properties are varied with rate of growth and percentage of summerwood.

Working stresses for extreme fiber in bending are varied with grade, extent of exposure, and size of piece; in horizontal shear, they are varied with grade; in compression parallel to grain, with grade, and exposure; in compression perpendicular to grain, with exposure.

Working stresses in shear are not varied with size or extent of exposure; in compression parallel to grain they are not varied with size; in compression perpendicular to grain they are not varied with grade or size; in modulus of elasticity they are taken as the same in all cases.

In Douglas fir and southern pine, working stresses for extreme fiber in bending, compression parallel to grain and compression perpendicular to grain are varied with rate of growth and percentage of summerwood. Values in these species in shear and modulus of elasticity are not varied with these properties.

Working values are given for three conditions of exposure during use: (*a*) Continuously dry, (*b*) Occasionally wet but quickly dried, (*c*) More or less continuously damp or wet. Judgment should be exercised as to the values to be used in a particular case.

> (*a*) Continuously dry contemplates use in interior construction, not subject to conditions of excessive dampness or high humidity.
>
> (*b*) Occasionally wet but quickly dried assumes use in such exterior structures as bridges, trestles, grandstands or bleachers, and exposed framework of open sheds.
>
> (*c*) More or less continuously damp or wet would apply to material exposed to waves or tidewater, or in contact with earth, or used in a building in portions that would be more or less continuously wet.

Grades for joist or beams may be used for members in direct tension, as in bottom chords of trusses, increase in size of defects toward ends being permissible because of the gradual application of stresses through splice plates or end connections.

For direct tension the same values as for extreme fiber stress in bending may be used. Straight grained wood has greater resistance to tension than to any other kind of stress. It has been found, however, practically impossible to design joints that will develop anywhere near the full tensile strength.

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[1] Published as information only.

Recognition of all loads in designing for moving loads, or loads concentrated near a support, gives an assumed shearing stress higher than is actually developed. In calculating the shear at one end of a beam, the concentrated loads between this end and a point distant three times the depth of the beam from the support may be considered as acting at this point. In moving loads, as on highway bridges or railway stringers, in computing the shear at one end it is safe to ignore all wheel loads between this support and a point three times the depth of the beam or stringer from this point when the balance of the span is assumed loaded so as to give a maximum shear stress.

The working values recommended may be used without allowance for impact up to 100 per cent.

When railway stringers are of two spans length, defects throughout the center two-thirds should be limited as in the center third of a single span stringer.

Timber constantly yields under long continued loading, acquiring a permanent set. This set with a fully loaded beam is about equal to the deflection using the modulus of elasticity as given in the tables. In order to minimize the results of sag, it is well to use values one-half those given in the table.

The working stresses for compression parallel to grain are for use on posts, struts, etc., with unsupported length not greater than eleven times their least dimension, as well as in end bearing on compression members. A short column or strut is more likely to fail at the end than at any other point in its length, and the variations in moisture content are greater there.

For columns with unsupported length eleven to twenty-two times their least dimension, working stresses should be reduced proportionately, from those given for compression parallel to grain in pieces whose length is eleven times their least dimension, to one-half these values in pieces whose length is twenty-two times their least dimension. For columns longer than twenty-two times their least dimension, working stresses should be determined by the Euler formula, using pin end conditions.

In long columns, stiffness is more of a factor than grade. Values of modulus of elasticity are average values and in long columns a factor of four should be applied to these values in order to obtain safe loads.

The influence of knots on the compressive strength of columns of constant cross-section decreases as the length increases. When the length reaches thirty times the least dimension, knots such as are allowable in the Select grade have practically no effect on the strength as a column.

WORKING STRESSES FOR DOUGLAS FIR AND SOUTHERN PINE

(Pounds per square inch)

Recommended by Forest Products Laboratory, United States Forest Service
for Grades Complying with

BASIC PROVISIONS FOR STRUCTURAL MATERIAL
OF
AMERICAN LUMBER STANDARDS

BEAMS AND STRINGERS
5 in. and Thicker

CONDITION OF USE	EXTREME FIBER IN BENDING, LB. PER SQ. IN.	COMPRESSION PERPENDICULAR TO GRAIN, LB. PER SQ. IN.	HORIZONTAL SHEAR, LB. PER SQ. IN.	MODULUS OF ELASTICITY, LB. PER SQ. IN.
CONTINUOUSLY DRY				
Douglas fir, Coast type:				
Dense Select	1750	380	90	1 600 000
Select	1600	345	90	1 600 000
Common	1200	325	72	1 600 000
Southern Pine:				
Dense Select	1750	380	110	1 600 000
Select	1600	345	110	1 600 000
Common	1200	325	88	1 600 000
OCCASIONALLY WET, BUT QUICKLY DRIED				
Douglas fir, Coast type:				
Dense Select	1515	265	90	1 600 000
Select	1385	240	90	1 600 000
Common	1040	225	72	1 600 000
Southern Pine:				
Dense Select	1515	265	110	1 600 000
Select	1385	240	110	1 600 000
Common	1040	225	88	1 600 000
MORE OR LESS CONTINUOUSLY DAMP OR WET				
Douglas fir, Coast type:				
Dense Select	1165	235	90	1 600 000
Select	1065	215	90	1 600 000
Common	800	200	72	1 600 000
Southern Pine:				
Dense Select	1165	235	110	1 600 000
Select	1065	215	110	1 600 000
Common	800	200	88	1 600 000

POSTS AND TIMBERS

6 by 6 in. and Larger

CONDITION OF USE	COMPRESSION PARALLEL TO GRAIN, LB. PER SQ. IN.	COMPRESSION PERPENDICULAR TO GRAIN, LB. PER SQ. IN.	MODULUS OF ELASTICITY, LB. PER SQ. IN.
CONTINUOUSLY WET			
Douglas fir, Coast type:			
Dense Select	1285	380	1 600 000
Select	1175	345	1 600 000
Common	880	325	1 600 000
Southern Pine:			
Dense Select	1285	380	1 600 000
Select	1175	345	1 600 000
Common	880	325	1 600 000
OCCASIONALLY WET, BUT QUICKLY DRIED			
Douglas fir, Coast type:			
Dense Select	1165	265	1 600 000
Select	1065	240	1 600 000
Common	800	225	1 600 000
Southern Pine:			
Dense Select	1165	265	1 600 000
Select	1065	240	1 600 000
Common	800	225	1 600 000
MORE OR LESS CONTINUOUSLY DAMP OR WET			
Douglas fir, Coast type:			
Dense Select	990	235	1 600 000
Select	905	215	1 600 000
Common	680	200	1 600 000
Southern Pine:			
Dense Select	990	235	1 600 000
Select	905	215	1 600 000
Common	680	200	1 600 000

JOIST AND PLANK
4 in. and Less in Thickness

CONDITION OF USE	EXTREME FIBER IN BENDING, LB. PER SQ. IN.	COMPRESSION PARALLEL TO GRAIN, LB. PER SQ. IN.	COMPRESSION PERPENDICULAR TO GRAIN, LB. PER SQ. IN.	HORIZONTAL SHEAR, LB. PER SQ. IN.	MODULUS OF ELASTICITY, LB. PER SQ. IN.
CONTINUOUSLY DRY					
Douglas fir, Coast type:					
Dense Select......	1750	1285	380	90	1 600 000
Select............	1600	1175	345	90	1 600 000
Common.........	1200	880	325	72	1 600 000
Southern Pine:					
Dense Select......	1750	1285	380	110	1 600 000
Select............	1600	1175	345	110	1 600 000
Common.........	1200	880	325	88	1 600 000
OCCASIONALLY WET, BUT QUICKLY DRIED					
Douglas fir, Coast type:					
Dense Select......	1350	1040	265	90	1 600 000
Select............	1235	950	240	90	1 600 000
Common.........	980	750	225	72	1 600 000
Southern Pine:					
Dense Select......	1350	1040	265	110	1 600 000
Select............	1235	950	240	110	1 600 000
Common.........	980	750	225	88	1 600 000
MORE OR LESS CONTINUOUSLY DAMP OR WET					
Douglas fir, Coast type:					
Dense Select......	1040	885	235	90	1 600 000
Select............	950	805	215	90	1 600 000
Common.........	750	640	200	72	1 600 000
Southern Pine:					
Dense Select......	1040	885	235	110	1 600 000
Select............	950	805	215	110	1 600 000
Common.........	750	640	200	88	1 600 000

In the report of the sub-committee for 1924, reference was made to a new type of flask which might be satisfactory for distilling high boiling oils such as creosote, road oils, fuel oils, etc. The flask illustrated in Fig. 1 is now being considered by the sub-committee, by Committees D-2 on Petroleum Products and Lubricants and D-4 on Road and Paving Materials, and by the preservative committees of the American Wood Preservers' Association and the American Railway Engineering Association. Comparative tests on creosote by the new flask and the present standard retort described in Section 11 of the Standard Methods of Testing Creosote Oil (D 38 – 24) indicate that results by the new flask are similar in value but more uniform, as between different operators. Additional comparative tests are now being made at eight representative laboratories, under the direction of the sub-committee. Creosote, petroleum and mixtures are included in these tests. In addition to these supervised tests, the committees mentioned above have requested that all persons interested in the analysis of creosote, road oils and tars, fuel oils, etc., try this flask in comparison with the distilling vessel now used, and communicate with the chairman of one of these committees, giving their results and

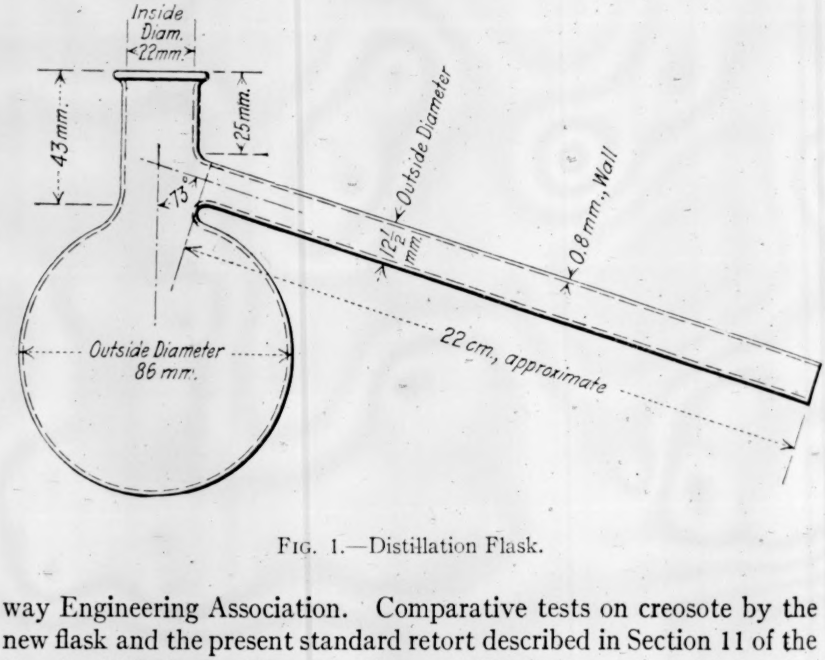

Fig. 1.—Distillation Flask.

opinions. In using the new flask, the set-up of distillation apparatus, including position of thermometer, condenser, etc., should be exactly as it is now specified in the various methods.

Representatives of Sub-Committee VI, in conference with representatives of Committee D-2 on Petroleum Products and Lubricants, have agreed on a plan for cooperative work with the American Wood Preservers' Association, for preparing methods of analysis for petroleum for use in creosote-petroleum mixtures.

Respectfully submitted on behalf of the sub-committee,

S. R. Church,
Chairman.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS

During the past year, the scope of work of Committee D-8 was materially broadened by action of the Executive Committee in changing its title from "Committee D-8 on Waterproofing Materials" to "Committee D-8 on Bituminous Waterproofing and Roofing Materials." The committee at once took active steps to increase its membership so as to be thoroughly representative of bituminous roofing interests from both producing and consuming standpoints and these new members were promptly assigned to the various sub-committees.

The committee's work on roofing products naturally centered in its Sub-Committee IV on Specifications (including Analytical Methods) for Membrane Materials. This sub-committee has been very active during the past year and due to its efforts Committee D-8 is in a position to recommend four new tentative specifications and one new tentative method of test to the Society. An additional tentative specification is also offered as the result of the work of Sub-Committee III on Investigations of Bituminous Grouts and Mastics. It is expected that next year the committee will be able to offer a number of new tentative definitions and methods of test which will originate in its Sub-Committees I on Nomenclature and Definitions and V on Specifications for Bituminous Coatings for Cold Application.

PROPOSED REVISIONS OF TENTATIVE METHODS OF TEST

1. *Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (D 147 - 24 T).*[1]—Through cooperation with Committee E-8 on Nomenclature and Definitions, the following revisions are recommended in these methods:

Section 1.—Change the definitions to read as follows by the addition of the italicized words and the omission of those in brackets:

"Bituminous Grout.—A mixture of bituminous material as a binder and sandy mineral matter as an aggregate, which [between 149° C. (300° F.) and 205° C. (400° F.)] *when heated to a suitable temperature* becomes sufficiently fluid to flow into place without mechanical manipulation, and which on cooling congeals to a compact mass."

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 1017 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 538.

"Asphalt Mastic.—(1) A mixture containing asphaltic material as a binder and graded mineral matter as an aggregate, or (2) pulverized native rock asphalt (to which asphaltic material may have been added); either of which [at approximately 205° C. (400° F.)] *when heated to a suitable temperature* may be poured into place but which requires a trowel to form it into a compact mass."

It is recommended that the methods as revised, be continued as tentative.

2. *Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 146 – 23 T).*[1]— Pending a more complete revision next year, the following additions are recommended in these methods in order to make them applicable to the proposed new tentative specifications for roofing materials:

Title.—After the word "Waterproofing" insert the words "*and Roofing.*"

Section 15.—Insert "(*a*)" before the present paragraph "Loss on Heating" and add the following new paragraphs:

"(*b*) *Extraction of Saturant. Coal-Tar Saturated Felt Only.*—From the rolls making up the sample, strips 6 in. wide shall be cut across the sheet. Sufficient of these shall be taken to make up a sample of 250 g. ±5.0 g. These strips then shall be rolled up and placed in the wire basket of the extraction apparatus described in the Tentative Methods of Testing Bituminous Mastics (Serial Designation: D 147 – 24 T) or other extraction apparatus of suitable construction. The sample shall then be covered with a disk of soft filter paper to insure an even distribution of the solvent. The extraction shall be carried out as described in Methods D 147 – 24 T.

"(*c*) *Recovery of Saturant from CS_2 Extract.*—The CS_2 extract containing the bitumen shall be transferred to a 500-cc., short-neck, round-bottom flask. The flask shall be equipped with a Hempel column, 16 cm. in length, 15 mm. in inside diameter and filled to a depth of 5 cm. with glass beads. The Hempel column shall be connected with a water cooled condenser. A 0 – 80° C. thermometer as described in the Tentative Method of Float Test for Bituminous Materials (Serial Designation: D 139 – 24 T) of the American Society for Testing Materials[2] shall be placed in the Hempel column so that the top of the bulb shall be opposite the bottom of the tubulature. Distillation for the removal of CS_2 shall be continued by means of a steam bath until drops cease to fall from the condenser. The distillation then shall be carried out over a small flame, heating carefully and observing the thermometer until the distillation ceases and the temperature of the vapor as indicated by the thermometer shows no further rise on slightly increasing the flame.

"(*d*) *Distillation of Saturant.*—When the contents of the flask is sufficiently cool to be poured without appreciable vaporizing, 100 g. ±0.1 g. shall be dis.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 796 (1923); also 1924 Book of A.S.T.M. Tentative Standards, p. 529.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 925 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 407.

tilled in accordance with the method described in Sections 10, 11 and 12 of the Standard Methods of Testing Creosote Oil (Serial Designation: D 38) of the American Society for Testing Materials.[1]

"Fractions shall be taken at 210° C. and 235° C. and the distillation stopped at that point. The per cent of distillate by weight at each temperature shall be calculated on the basis of the weight of sample taken for distillation."

It is recommended that the methods as revised be continued as tentative.

NEW SPECIFICATIONS AND METHODS OF TEST SUBMITTED AS TENTATIVE

The committee recommends for publication as tentative the following specifications and methods of test appended hereto.[2] If the proposed tentative specifications (*d*) and (*e*) are accepted it also recommends that they supersede the existing Tentative Specifications for Felted Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 172 – 23 T)[3] and that the latter be withdrawn from publication.

(*a*) Specifications for Acid-Resisting Asphalt Mastic.
(*b*) Specifications for Smooth-Surfaced Asphalt Roll-Roofing.
(*c*) Specifications for Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles.
(*d*) Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs.
(*e*) Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs.
(*f*) Methods of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles.

TENTATIVE SPECIFICATIONS ADVANCED TO STANDARD

No adverse criticism of the following tentative specifications have been received and the committtee therefore recommends that they be advanced to standard without change.

(*a*) Tentative Specifications for Asphalt Mastic for Use in Waterproofing (D 169 – 24 T).[4]
(*b*) Tentative Specifications for Bituminous Grout for Use in Waterproofing Above Ground Level (D 170 – 24 T).[4]

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[1] 1924 Book of A.S.T.M. Standards.

[2] See pp. 759–775 and 792.—ED.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 787 (1923); also 1924 Book of A.S.T.M. Tentative Standards, p. 520.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, pp. 1011, 1013 (1924); also 1924 Book of A.S.T.M. Tentative Standards, pp. 514, 516.

(*c*) Tentative Specifications for Bituminous Grout for Use in Waterproofing Below Ground Level (D 171 – 24 T).[1]

(*d*) Tentative Specifications for Asphalt for Use in Damp-proofing and Waterproofing Below Ground Level (D 40 – 24 T).[1]

(*e*) Tentative Specifications for Asphalt for Use in Damp-proofing and Waterproofing Above Ground Level (D 144 – 24 T).[1]

(*f*) Tentative Specifications for Primer for Use With Asphalt in Damp-proofing and Waterproofing Below and Above Ground Level (D 41 – 22 T).[2]

(*g*) Tentative Specifications for High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (D 42 – 24 T).[1]

(*h*) Tentative Specifications for High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level (D 145 – 24 T).[1]

(*i*) Tentative Specifications for High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (D 200 – 24 T).[1]

(*j*) Tentative Specifications for High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level (D 201 – 24 T).[1]

(*k*) Tentative Specifications for Creosote Oil for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing Below and Above Ground Level (D 43 – 22 T).[2]

In addition the committee recommends that the following two tentative specifications be advanced to standard with the revision indicated below which will bring them in conformity with the new tentative specifications which have been proposed.

(*l*) Tentative Specifications for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 173 – 23 T).[3]

(*m*) Tentative Specifications for Burlap Saturated with Bituminous Substances for Use in Waterproofing (D 174 – 23 T).[3]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, pp. 1015, 999, 1001, 1003, 1005, 1007, 1009 (1924); also 1924 Book of A.S.T.M. Tentative Standards, pp. 518, 498, 500, 504, 506, 508, 510.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, pp. 867, 873 (1922); also 1924 Book of A.S.T.M. Tentative Standards, pp. 502, 512.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, pp. 790, 793 (1923); also 1924 Book of A.S.T.M. Tentative Standards, pp. 523, 526.

In these two specifications it is recommended that the present Sections 13, 14 and 15 under the heading "VI Inspection and Rejection" be deleted and that the following new Section 13 be substituted therefor.

"VI INSPECTION

13. "Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract."

The results of letter ballot of the committee upon the recommendations made in this report are as follows:

Items	Affirmative	Negative	Not Voting
I. Proposed Revisions of Existing Tentative Standards			
Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (D 147 - 24 T)	26	0	23
Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 146 - 23 T)	25	0	24
II. Proposed New Tentative Standards			
Specifications for Acid-Resisting Asphalt Mastic	25	0	24
Specifications for Smooth-Surfaced Asphalt Roll Roofing	26	1	22
Specifications for Slate-Surfaced Asphalt Roll Roofing and Slate-Surfaced Asphalt Shingles	25	2	22
Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs	28	0	21
Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs	26	1	22
Methods of Testing Smooth-Surfaced Asphalt Roll Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles	28	0	21
III. Advancement of Tentative Standards to Standard			
Tentative Specifications for Asphalt Mastic for Use in Waterproofing (D 169 - 24 T)	24	1	24
Tentative Specifications for Bituminous Grout for Use in Waterproofing Above Ground Level (D 170 - 24 T)	24	1	24
Tentative Specifications for Bituminous Grout for Use in Waterproofing Below Ground Level (D 171 - 24 T)	24	1	24
Tentative Specifications for Asphalt for Use in Damp-proofing and Waterproofing Below Ground Level (D 40 - 24 T)	25	1	23
Tentative Specifications for Asphalt for Use in Damp-proofing and Waterproofing Above Ground Level (D 144 - 24 T)	27	1	21
Tentative Specifications for Primer for Use with Asphalt in Damp-proofing and Waterproofing Below and Above Ground Level (D 41 - 22 T)	26	1	22
Tentative Specifications for High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (D 42 - 24 T)	23	1	25
Tentative Specifications for High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level (D 145 - 24 T)	25	1	23
Tentative Specifications for High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (D 200 - 24 T)	23	1	25
Tentative Specifications for High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level (D 201 - 24 T)	25	1	23
Tentative Specifications for Creosote Oil for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing Below and Above Ground Level (D 43 - 22 T)	22	0	27
Tentative Specifications for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 173 - 23 T)	22	1	26
Tentative Specifications for Burlap Saturated with Bituminous Substances for Use in Waterproofing (D 174 - 23 T)	22	1	26

This report has been submitted to letter ballot of the committee, which consists of 49 members, of whom 27 have voted affirmatively, 1 negatively, and 21 have refrained from voting.

Respectfully submitted on behalf of the committee,

S. T. WAGNER,
Chairman.

PRÉVOST HUBBARD,
Secretary.

EDITORIAL NOTE

The proposed revisions of the Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures; and of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing were accepted. The tentative methods as thus revised appear on pages 786 and 776, respectively.

The Tentative Specifications listed in this report as items (*a*) to (*m*), pages 344 and 345, were approved at the annual meeting and subsequently adopted by letter ballot of the Society on August 28, 1925, and appear in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

The proposed new Specifications for Acid-Resisting Asphalt Mastic; for Smooth-Surfaced Asphalt Roll-Roofing; for Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles; for Asphalt-Saturated Roofing Felt for Use in Waterproofing and Constructing Built-up Roofs; and for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-up Roofs; and Methods of Testing Smooth-Surfaced Asphalt Roll Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles were accepted for publication as tentative and appear on pages 759–775 and 792.

The withdrawal of the Tentative Specifications for Felted Fabrics Saturated with Bituminous Substances for Use in Waterproofing was approved.

REPORT OF COMMITTEE D-9
ON
ELECTRICAL INSULATING MATERIALS

The committee held three meetings during the year as follows in addition to the one held during this annual meeting of the Society:

At Pittsfield, October 16–17, 1924;
At New York, January 23, 1925;
At Philadelphia, March 27, 1925.

The activities of the committee are still confined to the improvement of methods of testing which it has already standardized and to the development of additional methods.

EXISTING STANDARDS AND TENTATIVE STANDARDS

There are before the Society two standards and seven tentative standards dealing with electrical insulating materials. The committee recommends the following action with respect to them:

Standard Methods of Testing Molded Insulating Materials (*D 48 – 24*)[1].—Experience has shown that the procedure for making the various tests at elevated temperatures was inadequate and permitted a considerable variation in results. It is not practicable in commercial testing to make sufficiently precise tension tests at high temperatures and as the desired information is, for all practical purposes, given by a transverse test, it is believed that tension tests at elevated temperatures could be omitted. After considerable experimental work and study by a number of laboratories, a more precise procedure has been agreed upon for compression, transverse and dielectric strength tests at elevated temperatures.

It has been found that the various tests now prescribed after immersion in water for 48 hours are used very little, consequently it is felt that all such tests, except those for dielectric strength, could be omitted.

General experience indicates that a specimen of cylindrical shape is better adapted for the compression test than a cube, as prescribed at present. Furthermore, the height of the present specimen is insufficient because the plane of cleavage does not always break through the side of the specimen. A cylindrical specimen is therefore proposed with an increase in height.

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[1] 1924 Book of A.S.T.M. Standards.

Some criticism has been made of the water absorption specimen which is the test cup used for the dielectric strength test with the rim sawed off. It is proposed that this specimen be replaced with the compression test specimen. Furthermore, the committee now believes that 100 hours immersion as at present prescribed is unnecessarily long and that 48 hours is sufficient.

In accordance with these proposals, and the need for greater clarity in certain minor points, the following revisions of Methods D 48 – 24 are recommended for publication as tentative:

Section 2.—Change the first sentence to read as follows by the addition of the italicized words:

"Any standard testing machine may be used *provided the error in the loading range does not exceed 1 per cent.*"

Section 3.—Change the second sentence to read as follows by the addition of the italicized word and the omission of those in brackets:

"It shall be molded in a hardened [and ground] steel mold *ground* to the dimensions given in Fig. 1."

Section 4.—Omit the second and third paragraphs reading as follows:

"Three specimens shall be tested after heating in an oven for one hour at a temperature which is 10° C. (18° F.) below the distortion point of the material, as determined in accordance with Sections 18 to 21. Each specimen shall be taken from the oven and tested immediately while hot.

"Two specimens shall be tested after they have been entirely immersed in water for 48 hours at normal room temperature. The specimens shall be pulled apart at normal room temperature of about 20° C. (68° F.) after the surface water has been removed by wiping with a dry cloth."

Change the first sentence of Paragraph (*b*) from its present form: namely,

"The test specimen shall be pulled apart at such a speed that the beam can be kept well balanced."

to read as follows:

"The speed of the head of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. (1.27 mm.) per minute with the machine running idle."

Fig. 2.—Change from a cube to a cylinder having a height of 1.25 (3.18 cm.) in. and a diameter of 1.125 in. (2.86 cm.) Omit the words "Manufacturer's Name and 'Test Specimen No. 3' Molded on Top in Small Round Body Raised Letters."

Section 6.—Change from its present form: namely,

"Any standard testing machine may be used. The pressure head used for standard compressive strength tests of cement blocks is satisfactory for this purpose. A sheet of soft annealed copper about $\frac{1}{32}$ in. (1 mm.) thick, or heavy blotting paper, shall be placed above and below the specimen to equalize irregularities."

to read as follows:

"Any standard testing machine may be used provided it is accurate to within 1 per cent for the lowest load for which it is used. One end of the specimen shall bear upon an accurately centered spherical bearing block, located whenever practicable at the top and the metal bearing plates shall be directly in contact with the ends of the test specimen. The pressure heads used for standard compression tests of cement (see Tentative Specifications and Tests for Compressive Strength of Portland-Cement Mortars (Serial Designation: C 9 - 16 T) of the American Society for Testing Materials) are satisfactory for this test."

Section 7.—In the first sentence, change the word "cube" to "cylinder." Add a new sentence to read as follows:

"The ends shall be flat and perpendicular to the axis, being ground to this condition if necessary."

Section 8.—Omit the second and third paragraphs, reading as follows:

"Three specimens shall be crushed after heating for one hour at a temperature which is 10° C. (18° F.) below the distortion point of the material as determined in accordance with Sections 18 to 21. Each specimen shall be taken from the oven and tested immediately while hot.

"Two specimens shall be crushed after immersion in water at normal room temperature for 48 hours, with all surface water wiped off with a dry cloth."

Insert new paragraphs to read as follows:

"Additional tests shall be made at elevated temperatures, the actual temperatures selected depending upon the use that is to be made of the material. Five specimens shall be tested at each temperature selected. The specimen shall be kept at that temperature a sufficient length of time to become of uniform temperature throughout and shall be maintained at that temperature during the test.

"NOTE 1.—A convenient method of making this test is to submerge the specimens in a suitably lagged, electrically heated oil bath of about two gallons capacity. The specimens should be placed in the bath at least one-half hour before testing and the temperature maintained approximately constant at the desired testing temperature until the completion of that test. A pale mineral oil having a viscosity of 100 seconds ± 10 seconds at 38° C. (100° F.) Saybolt is satisfactory for this purpose. The specimens should not be placed in the oil an excessive time in advance of testing. The oil bath should be well stirred to insure uniform temperature throughout. The temperature should be determined by means of a thermocouple sealed in a similar specimen in the bath; or by the use of an A.S.T.M. partial immersion thermometer conforming to the Tentative Specifications for Partial-Immersion Thermometer for General Use, −20 to +150° C., 0 to +300° F. (Serial Designation: D 182 - 23 T) of the

American Society for Testing Materials,[1] so inserted in a hole in a specimen that the bulb is completely enclosed and sealed in.

"If tests are required over the whole range of temperature rather than at particular points, the method of testing may be found simpler and quicker if modified in accordance with Note 2, below.

"NOTE 2.—The oil with 5 specimens immersed in it should be heated to the highest temperature to be used and allowed to cool slowly. Specimens should be tested in succession and the average temperature during the actual time of testing of each specimen taken as that corresponding to each strength determination. Specimens should be added to the bath successively on such a schedule that each will remain in the bath at least ½ hour before testing. An average curve shall be drawn through the plotted data."

Change the first sentence of Paragraph (*b*) from its present form: namely,

"The load shall be applied in a direction at right angles to that in which the pressure was applied in molding, and at such a rate of speed that will permit the beam to be kept well balanced from zero load until the specimen is crushed."

to read as follows:

"The load shall be applied on the ends of the specimen and the cross-head speed of the testing machine shall be such that the beam of the machine can be kept balanced but shall not exceed 0.050 in. (1.27 mm.) per minute when the machine is running idle."

In Paragraph (*e*), change the word "jaws" to read "head." Add a new sentence to read as follows:

"The time required for testing each specimen should be recorded."

Section 10.—Change to read as follows:

"Any standard testing machine may be used provided it is accurate to within 1 per cent for the lowest load for which it is used. The distance between points of support shall be 4 in. (10.16 cm.). The supports shall have the contact edges rounded to a radius of ⅛ in. (3.18 mm.). The load shall be applied midway between the supports by a pressure piece, the bearing edge of which shall be rounded to a radius of ⅛ in. (3.18 mm.)."

Section 12.—In the first sentence of Paragraph (*a*) change the words "four specimens" to read "five specimens."

Change the second paragraph from its present form: namely,

"Three specimens shall be tested after immersion in water at normal room temperature for 48 hours with all surface water wiped off with a dry cloth."

to read as follows:

"Additional tests shall be made at elevated temperatures as outlined in Section 8."

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 905 (1923); also 1924 Book of Tentative Standards, p. 677.

Change the first two sentences of Paragraph (*b*) from their present form: namely,

> "The load shall be applied at as slow a speed as possible, so that the beam may be kept well balanced from zero load until the first sign of failure. All tests shall be made at room temperature of about 20° C. (68° F.)."

to read as follows:

> "The cross-head speed of the testing machine shall be such that the beam of the machine can be kept balanced but shall not exceed 0.050 in. (1.27 mm.) per minute when the machine is running idle."

Section 13.—Change Paragraph (*e*) to read as follows by the addition of the italicized word and the omission of those in brackets:

> "The [amount of] *maximum* deflection at the center in inches or in millimeters."

Omit Paragraph (*f*) reading as follows:

> "The distance between the supports in inches or in millimeters."

Section 16 (*b*).—Change the second paragraph from its present form: namely,

> "Three specimens shall be punctured after heating one hour in an oven heated to 10° C. (18° F.) below the distortion point of the material as determined in Sections 18 to 21. The specimen shall be taken from the oven and immediately punctured in air while hot. This test is intended to cover all molded materials which do not withstand working temperatures above 125° C. (257° F.). Tests on materials which resist high temperatures may be made above 125° C. (257° F.) when required."

to read as follows:

> "Additional tests shall be made at elevated temperatures, the actual temperatures selected depending upon the use that is to be made of the material. Five specimens shall be tested at each temperature while in an oven maintained at the proper temperature, either by hand adjustment or by automatic thermostat. Specimens shall be placed in the oven at least ½ hour previous to testing and the temperature shall then be maintained approximately constant at the testing temperature until the completion of that test. Temperature shall be determined by means of a thermocouple or a mercury thermometer placed in the oven close to the specimens and care shall be taken to avoid placing the specimens too close to the heater.
>
> "Specimens with which arcing over the edge is encountered shall be tested under oil as described under Section 16, and in such cases the oil bath containing the specimens shall be installed in the oven and the temperature determined by immersing the thermometer in the oil."

In the third paragraph, change the words "Two specimens" to "Three specimens."

Section 17.—Add a new paragraph to read as follows:

> "(*e*) Temperature at which each test was made."

Section 23.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"The test specimen [No. 1] *No. 3* shown in [Fig. 4] *Fig. 2* shall be used for this test. [After the puncture tests prescribed in Section 16 (*b*) are completed on the five specimens, three of them shall be taken and the entire rim of each specimen shall be sawed off with a hack saw $\frac{1}{4}$ in. (6 mm.) back from the top edge of the specimen so as to expose a more or less uniformly cut surface.]"

Section 24.—Change the first sentence from its present form: namely,

"Weigh each of the three test specimens after the rim has been sawed off."

to read as follows:

"Three specimens shall be tested. Weigh each of the three separately in the as received condition."

In the fifth and sixth sentences change the words "100 hours" to "48 hours."

Section 25.—In paragraphs (*c*) and (*d*) change the words "100 hours" to "48 hours."

Tentative Methods of Testing Insulating Varnishes (D 115–24 T).[1]—The committee has found that the procedure for determining viscosity, where the MacMichael viscosimeter is used, needs to be more precisely outlined.

The procedure for making the dielectric strength test should of course be in accordance with that prescribed in the Tentative Methods of Testing Sheet and Tape Insulating Materials for Dielectric Strength. It is therefore recommended that the tentative methods be continued as tentative but revised as follows:

Section 3.—Change to read as follows:

"(*a*) The viscosity shall be determined with a Stormer or a MacMichael viscosimeter. For the Stormer instrument, it shall be stated in terms of the viscosity of distilled water determined with the same instrument and under the same conditions. For the MacMichael viscosimeter, the viscosity shall be stated in absolute units, the centipoise (cp.). The short-tube type of efflux viscosimeter usually employed for lubricating oils is not acceptable. The temperature of the varnish shall be 20° C. (68° F.).

"(*b*) The MacMichael viscosimeter shall be calibrated as follows:

"Obtain fair sized samples (2 to 5 gal. or more) of several oils of suitable viscosities. These should be selected so that they fall in the ranges of viscosity in which measurements are likely to be made. This is to permit calibration of the instrument with an oil of the same order of viscosity as the products that are to be tested. A convenient series of samples for the testing of insulating varnish is:

> "Mineral Oil having an absolute viscosity of approximately 20 centipoises at 20° C. (approximately 65 seconds, Saybolt Universal Viscosity, at 37.8° C.).

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 1023 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 544.

"Mineral Oil having an aboslute viscosity ot approximately 40 centipoises at 20° C. (approximately 100 seconds, Saybolt Universal Viscosity, at 37.8° C.).

"Mineral Oil having an absolute viscosity of approximately 170 centipoises at 20° C. (approximately 300 seconds, Saybolt Universal Viscosity, at 37.8° C.).

"Castor Oil having an absolute viscosity of approximately 992 centipoises at 20° C. (approximately 110 seconds, Saybolt Universal Viscosity, at 98.9° C.).

"Retain the major portion of each of these samples and send 4 to 8 oz. to the Bureau of Standards, Washington, D. C., for determination of absolute viscosity. Specify 20° C. as the temperature at which measurements are to be made. The samples, if they are properly refined filtered oils, are regarded as stable for at least one year if kept uncontaminated in closed opaque containers.

"NOTE.—Standardized samples of known absolute viscosity may be obtained directly from the Bureau."

"(*c*) When using the MacMichael viscosimeter, it is necessary (1) to obtain the zero adjustment prior to placing the varnish in the cup, (2) to adjust to a sufficiently low speed to prevent eddy currents.

"(*d*) The report shall include such details as the kind of instrument used, the size of the counterweight if a Stormer instrument is used, or the size of the wire in case the MacMichael instrument is used, etc."

Section 8.—Change the phrase in Paragraph (*c*) reading "in free air" to read "in dust-free air."

Change Paragraph (*d*) by adding the words "in dust-free air" after the word "baked".

Section 9 (*a*).—Change the second sentence from its present form: namely,

"The potential shall be applied at a low value and gradually raised at a rate of 0.5 kv. per second."

to read as follows:

"Starting at zero, the voltage shall be increased uniformly to breakdown at a rate of 0.5 kilovolt per second, except that if breakdown occurs at this rate in less than 40 seconds, the rate shall be decreased so that breakdown will occur in not less than 40 seconds. If the material fails at less than 5 kilovolts, the minimum time shall be reduced from 40 seconds to 20 seconds."

Tentative Methods of Testing Cable Splicing and Pothead Compounds (D 176 - 24 T).[1]—Cable splicing compounds which are soft at normal temperatures are now being extensively used. Since with these compounds the danger of the formation of voids between electrodes, when the material cools in the specimen container, is practically nil, the need for using spherical electrodes does not exist. It is therefore recommended that in the case of soft compounds the standard

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 1046 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 593.

1-in. disk electrodes used for testing transformer oil be prescribed as a matter of simplification and convenience.

It is recommended that the methods be continued as tentative with the following revisions:

Section 6.—Change to read as follows:

"(*a*) In the case of materials which are hard at room temperature, they shall be tested between hemispherical electrodes embedded therein which are ½ in. in diameter separated by a gap of 0.1 in.

"Note.—A convenient form of apparatus for holding the electrodes and compound is described in the appendix.

"(*b*) In the case of materials which are soft at room temperature, they shall be tested between polished brass or copper circular disk electrodes, 1 in. in diameter and having a square edge, the electrodes to be placed with their axes horizontal and coincident and with a gap of 0.1 in. between their adjacent faces.

"Note.—This test can be made with the apparatus normally used for testing transformer and switch oils. (See the Standard Methods of Testing Transformer and Switch Oils (Serial Designation: D 117) of the American Society for Testing Materials.[1])

Tentative Methods of Testing Untreated Insulating Paper (D 202 – 24 T).[2]—Section 12 (*c*) is not correctly worded. It is recommended that it be changed to read as follows:

"The machine shall be graduated to read 1 lb. or 1 kg. or less per scale division for testing paper breaking at 22.7 kg. (50 lb.) or over and to 0.5 lb. or 0.5 kg. or less for testing paper breaking under 22.7 kg. (50 lb.)."

It is further recommended that a new test, to determine the permeability of paper to air, be added as follows:

X. AIR RESISTANCE

"32. *Apparatus.* (*a*)—The instrument shall consist of two aluminum open-top cylinders, one of which is inverted and slides into the other which is fixed. The movable cylinder shall be provided with a circular aperture in the closed end and a flat ring clamp for holding the paper specimen across this aperture.

(*b*) The fixed cylinder shall be 25.4 cm. (10 in.) in height and shall have an external diameter of 8.60 cm. (3.38 in.) and an internal diameter of 8.25 cm. (3.25 in.). Four slender bars, each 18.5 cm. (7.3 in.) long, 3 mm. (0.12 in.) wide and approximately 1.5 mm. (0.06 in.) thick shall be mounted vertically and equi-distantly on the inner surface of the fixed cylinder to act as guide tracks for the movable cylinder. The movable cylinder shall be graduated in units of 50 cc. and shall have a total range of 350 cc. It shall be 25.4 cm. (10 in.) high and shall have an external diameter of 7.62 cm. (3.00 in.) and an internal diameter of 7.35 cm. (2.90 in.). It shall weigh, including the flat ring clamp and the two knurled nuts, 567 g. (20 oz.) ± 5 g. (0.18 oz.). The movable cylinder and the flat ring clamp shall have a concentric circular aperture of 6.44 sq. cm. (1.00 sq. in.) in area. When this aperture is too large for the specimen a similar movable cylinder and flat ring clamp having a concentric circular aperture of 1.61 sq. cm. (0.25 sq. in.) in area shall be used.

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[1] 1924 Book of A.S.T.M. Standards.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 1051 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 598.

"NOTE.—Results obtained with apertures of different areas are not directly proportional to the areas of the apertures. The proportionality factor must be determined by experiment.

"33. *Specimen.*—The specimens shall be not less than 3.5 cm. (1.36 in.) nor more than 5.1 cm. (2.0 in.) in width cut from the original samples, taken according to Section 2 (*a*) and shall be as long as the width of the original roll of paper. If the specimens are selected from pads of tape they shall be not less than 2.1 cm. (0.81 in.) nor more than 5.1 cm. (2.0 in.) wide and at least 30 cm. (12 in.) long. There shall be as many specimens as there are original samples.

"34. *Procedure.*—The fixed cylinder shall be placed on a rigid support so that its sides are vertical. Ethylene glycol shall be placed in it to a depth of 12.7 cm. (5 in.). The specimen (one thickness only) shall be secured tightly under the clamp on the movable cylinder completely covering the aperture. The movable cylinder is then floated on the ethylene glycol. The time required for the displacement of a certain amount of air is noted with a stop-watch. If possible, the amount of air displaced should be such that the time of displacement is not less than 20 seconds. The apparatus with its content of ethylene glycol shall be at the temperature of the conditioning room when the readings are taken. The time in seconds required for the displacement of 100 cc. (6.1 cu. in.) through a circular area (one side only) of 6.44 sq. cm. (1 sq. in.) of the paper is known as the air resistance of the paper.

"NOTE.—The clamp shall be tested for leakage by substituting a piece of tin foil 0.05 mm. (0.002 in.) in thickness for the paper and testing in the manner described above. When so tested, the leakage shall not exceed the rate of 50 cc. in five hours.

"The proper procedure for clamping the specimen or tinfoil is to turn both knurled nuts down onto the clamp simultaneously. If only one nut at a time is turned down, the clamp will not fit flat on the specimen and will consequently have an avoidable leak.

"Ethylene glycol is used in preference to distilled water because it does not corrode aluminum whereas distilled water does.

"35. *Report.*—The report shall include (1) the number of seconds required for the displacement of 100 cc. (6.1 cu. in.) of air; (2) the area of paper through which the air was displaced; (3) the thickness of the paper as obtained under Section 11 and (4) the room temperature."

The new material is to appear as Sections 32 to 35 and the present Sections 32 to 35 are to be renumbered accordingly.

It is recommended that this tentative standard as revised be continued in its present status.

Tentative Method of Testing Electrical Insulating Materials for Voltage Effects at Radio Frequencies (D 175 – 23 T).[1]—The committee finds that the flashover test in this method is not well suited for the purpose for which it is intended and is not being used. It is therefore recommended that subdivision (*B*), Sections 13 to 18, inclusive, be eliminated together with references to this test in other parts of the tentative standards.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 841 (1923); also 1924 Book of A.S.T.M. Tentative Standards, p. 586.

A high frequency dielectric endurance test which will be of more practical value and which can be substituted for the flashover test is being studied, but the committee is not yet prepared to make a definite recommendation.

In regard to the remaining standard and the three remaining tentative standards, it is recommended that they be continued in their present status.

PROPOSED NEW TENTATIVE STANDARD

Proposed tentative methods of testing laminated sheet insulating materials are appended to this report.[1] It is recommended that they be accepted for publication as a tentative standard. These methods only include at this time tests for tensile strength, transverse strength, dielectric strength at commercial frequencies and power factor and dielectric constant at radio frequencies, the electrical tests being the same as those already standardized for other materials. The development of further tests is under way.

MISCELLANEOUS

Insulating Varnishes.—Reference was made in the report of last year to an investigaton then under way of the suggested greater suitability of kraft paper as the base for dielectric strength test specimens. Comparative tests were made by six laboratories on two kinds of varnish to determine the relative value of copper and kraft paper as the base material for these specimens. The results indicated that although the variations among themselves in any one laboratory were considerably less with kraft paper than with copper, the variation among the average results reported by the different laboratories was of the order of 15 per cent for kraft paper and 20 per cent for copper. As a result of this investigation the present specification for the preparation of the test specimen was rewritten to provide for the use of kraft paper. But coincidentally with this work there has been considerable activity in the testing of varnish films (apart from a base) for dielectric strength, heat endurance, effect of water, oils, acids, etc., following the work begun by Harley A. Nelson as recorded in the Society's Proceedings.[2] The results thus far obtained give such promise that the activities of the Sub-Committee on Insulating Varnishes are being concentrated in this direction. In the meantime the suggested change in the existing specification for the preparation of the test specimen is being held up pending the results of this work on varnish films.

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[1] See p. 825.—ED.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 1111 (1921); Vol. 23, Part I, p. 290 (1923); Vol. 23, Part II, p. 356 (1923).

Experimental work is being continued by the sub-committee to determine the feasibility of a measurement for dielectric strength and insulation resistance of varnish in the liquid state. It would not be proposed to substitute such a test for the dielectric strength test of a varnish in film form; it would be an added test for the purpose of detecting the presence of moisture or impurities in the varnish before it is applied.

Molded Insulating Materials.—Some comment has been made on the use of the molded cup for dielectric strength tests. A disk has been used to some extent for this purpose, instead of the cup, with satisfactory results not only from the standpoint of dielectric strength but also as a means of judging the moldability of the material which was an important reason for the adoption of the cup specimen. However, a disk shape specimen is not suitable for cold molded materials. The sub-committee dealing with this class of materials is investigating this subject further.

Progress has been made in the development of an impact testing machine suitable for testing molded insulating materials to which reference was made last year. Three machines have been constructed by members of the sub-committee in accordance with a design which was agreed upon, and some preliminary tests have been made. Enough data have not yet been obtained to justify making recommendations at this time. The machines in question are of small capacity and of the Izod type, but readily converted to the Charpy type. In addition to these specially constructed machines, other machines of this same general type are being investigated.

An impact-fatigue test has also been considered but as no suitable machine is available on the market, the committee is dropping the matter for the present, as its commercial importance does not seem to be sufficient to justify the development of a special machine.

Some further attention has been given to the matter of high frequency testing. Considerable interest has been shown in this subject which, on account of the rapid developments in radio, has assumed importance. An additional method for measuring dielectric constant and phase difference, which is considered a better working method though not as precise as the existing one (Tentative Methods of Testing Electrical Insulating Materials for Phase Difference (Power Factor) and Dielectric Constant at Radio Frequencies (D 150-23 T)), has been discussed. Some changes in existing Methods

D 150 – 23 T are under consideration, particularly in connection with the water immersion test, the effect of humidity and the type of electrodes, but the committee is not prepared to make definite recommendations at this time.

Tests for surface insulation resistance and for volume resistivity have been given some attention during the year. It is very difficult, if not impossible, to separate surface leakage from volume leakage in the case of many commercial insulating materials when they are tested under relatively high humidity conditions. This makes it extremely difficult to prepare a test method for measuring surface resistance by itself, at least under such conditions. However, the matter is being investigated further by the committee although it has been considered of less importance than some of the other matters being studied.

Porcelain.—As the result of a large amount of experience in the testing of porcelain which has recently been made available to the committee, the indications are that the existing Tentative Methods of Testing Electrical Porcelain (D 116 – 24 T) will be radically revised during the coming year.

Sheet Insulating Materials.—A procedure for testing sheet materials for conducting particles has been prepared and discussed. While the procedure has gone through three revisions, it is not yet ready for submission.

An acidity test for untreated paper to be used for insulating purposes has received a great deal of attention on the part of one of the sub-committees. Great difficulty is being experienced in developing a reliable method for measuring the extremely small quantities of acid found in these materials. Two series of inter-laboratory tests have been made. In the first series, tests of six samples of paper were made by eight laboratories using three different methods. The results were not concordant, indicating the necessity for prescribing with great detail the exact procedure to be followed in all of the methods. The second series was made by nine laboratories using four samples of paper and eight different test methods. The data from these tests are now being collected and analyzed.

The subject of deterioration of sheet insulating materials is of course an exceedingly important one. As indicated in previous reports, the committee is endeavoring to develop a standard method for determining the rate of deterioration, but the necessary work which must first be done makes such a formidable program that progress has been slow, particularly as there were other problems which gave greater promise of more immediate results. However, the committee has organized a definite program and various laboratories have accepted investigation consignments under this program, the first part of which is the collection of available data on the effect of various deteriorating agencies on various kinds of sheet insulating materials. These deteriorating agencies will include ozone, oxygen, heat, electrical tension, moisture, acidity and catalytic agents, oils in compounds, chemical agents, light and mechanical stresses. A study of these data will indicate along what lines further experimental work needs to be done.

The thermal conductivity apparatus referred to in the report of the committee last year has been constructed by one of the laboratories represented on the committee and considerable work has been done with it. The results appear to be satisfactory, but before considering it for standardization, the results are to be checked by other laboratories. The principle involved is the one now generally accepted as the proper one for such measurements, namely, the measurement of the heat supplied to a definite area of a uniformly heated hot plate on one side of the specimen, the other side of the specimen being in contact with a similar plate which is kept at a low temperature by means of circulating water.

Transformer Oil.—As indicated in the past reports of the committee, a great deal of attention has been paid to the subject of deterioration of transformer oils. A vast amount of work has been and is being done on this problem which is of so great importance to the electrical industry. However, despite all of this work the committee is not yet in position to formally recommend a definite procedure for this test. Confirming the report of last year, the procedure first described in the Appendix to the 1923 report of Committee D-9 is giving the most promising results. A considerable number of these machines of the latest type which include changes resulting from previous experience are under trial in a number of laboratories. The committee has not, however, arrived at the point where it is prepared to make a definite recommendation in view of the importance of this matter and the large number of methods already in use. The committee feels that it must have an unusual amount of concrete evidence to justify standardizing any one procedure. It is hoped, however, that this evidence will be available in sufficient amount to justify action next year. In the meantime it desires to publish as an Appendix to this report, a revised description of this procedure. It includes certain refinements which experience has indicated necessary and new photographs of the apparatus as now constructed.

The subject of transformer oils has come up for international consideration through the International Electrotechnical Commission which last year appointed an Advisory Committee on transformer and other insulating oils. A meeting of a number of the Advisory Committees of the Commission, including this new one on transformer oils, was held at The Hague in April of this year. In view of the special importance of this subject in this country, the United States National Committee of the I.E.C., which is of course represented on this Advisory Committee, deemed it necessary to present at The Hague meeting a well prepared report which would represent the views of all interests in this country on this subject. The president of the United States Committee, recognizing that the A.S.T.M. had done more work in this particular field than any other organization, formally requested the Society to appoint a special representative committee to prepare a report, for the use of the United States Committee, on this subject. Such a committee was organized under the chairmanship of C. E. Skinner with the personnel of Committee D-9 prominently represented. The report which was prepared by this Special Committee and transmitted to the United States Committee, recommended that no attempt be made to prepare commodity specifications at this time but that an attempt be made to get international standardization of methods of testing. It further recommended that the test methods of this Society for transformer and switch oils be offered for international adoption with practically no change.

A member of Committee D-9, E. A. Snyder, who has made a special study of transformer oils and particularly of sludging tests thereof, was present at The Hague meeting as an official delegate of the United States Committee accredited to this Society. Mr. Snyder presented at this meeting a paper on sludging tests for transformer oils which contains a critical discussion of all of the various methods which are in use or have been proposed.

The following is the result of the letter ballot vote of the committee, consisting of 44 members, on the recommendations made in this report:

Item	Affirmative	Negative	Not Voting
I. PROPOSED REVISION OF STANDARD			
Standard Methods of Testing Molded Insulating Materials (D 48 - 24)	27	0	17
II. PROPOSED REVISION OF TENTATIVE STANDARDS			
Tentative Methods of Testing Insulating Varnishes (D 115 - 24 T)	28	1	15
Tentative Methods of Testing Cable Splicing and Pothead Compounds (D 176 - 24 T)	27	0	17
Tentative Methods of Testing Untreated Insulating Paper (D 202 - 24 T)	26	1	17
Tentative Methods of Testing Electrical Insulating Materials for Voltage Effects at Radio Frequencies (D 175 - 23 T)	29	0	15
III. PROPOSED NEW TENTATIVE STANDARD			
Proposed Tentative Methods of Testing Laminated Sheet Insulating Materials	27	0	17

This report has been submitted to letter ballot of the committee, which consists of 44 members, of whom 33 have voted affirmatively, none negatively, and 11 have refrained from voting.

Respectfully submitted on behalf of the committee,

F. M. FARMER,
Chairman.

H. S. VASSAR,
Secretary.

EDITORIAL NOTE

The proposed revisions of the Standard Methods of Testing Molded Insulating Materials were accepted for publication as tentative and appear on page 912.

The proposed revisions of the Tentative Methods of Testing Insulating Varnishes; of Testing Cable Splicing and Pothead Compounds; of Testing Untreated Insulating Paper; and of Testing Electrical Insulating Materials for Voltage Effects at Radio Frequencies were accepted. The tentative methods as thus revised appear on pages 797, 810, 815 and 804, respectively.

The proposed new Methods of Testing Laminated Sheet Insulating Materials were accepted for publication as tentative and appear on page 825.

APPENDIX

LIFE TEST FOR TRANSFORMER OILS

By E. A. Snyder[1] and D. C. Cox[1]

Note.—This method was described in Appendix I to the 1923 report of Committee D-9, and also in Appendix I to the 1924 report.[2] The following is a revision of the previous descriptions. It includes improvements that have been made in the construction of the apparatus as the result of further experience in several laboratories.

Apparatus

Referring to Figs. 1 and 2, *A* is a double-walled oven made of two sections of shellac-paper cylinders, the space between the cylinders being filled with asbestos. The top and bottom are made of asbestor lumber. The dimensions of the oven are approximately 22 in. in outside diameter by 13 in. deep. *L* is a removable asbestos lumber lid having a small hole, *C*, in the top for inserting the pipette to withdraw specimens of the samples. The lid also has a peep-hole, *E*, through which can be seen an index plate to determine when the different beakers during the course of rotation in the oven are properly located under the hole *C*. This peep-hole has a pivot cover which should be kept closed when not in use. To the under side of the lid is attached a steel condensation plate, slightly less in diameter than the inside of the oven and coned slightly so that condensed vapors, if any are formed, may drip safely beyond the edge of the beakers and thus eliminate the danger of contaminating the oil samples. *M* is a $\frac{1}{4}$-h-p., 220-volt, 2-phase, 1800 r.p.m., constant-speed motor, directly connected to the horizontal worm shaft with a flexible coupling, *F*. This horizontal shaft is supported by ring oil bearings, *O*. The worm gear shaft, *G*, is vertical and is driven by a 240 tooth gear which meshes with the single thread worm gear on the horizontal shaft. The vertical shaft is supported on a thrust bearing at the bottom end. The upper end is supported with a composition brass and graphite bearing which does not require oiling. *T* is a thermostat, the bulb of which is located horizontally above the beakers in a plane just slightly above the top of the asbestos-covered steel center chimney. This chimney, supported centrally in the oven, induces a vertical current of warm air from the heating unit. Arriving at the top of the oven, the air current turns radially and downward, bathing the sample beakers on its way. The thermostat, when contact is made in it, energizes the coil of the relay, *X*. Two sliding contacts (*W* and *Z*), are connected to the make and break of the relay. If the oven temperature falls, the thermostat contact is made, the relay operates, thereby short circuiting the two sliding contacts thus decreasing the external resistance in the heater circuit and increasing the current. The amount of current flowing in the heating unit may be set at any suitable figure by means of the rheostat, *Y*, and the relay, *X*, as noted below under "Adjustment of Apparatus." The heater is in one unit in the form of a 3-turn helix, freely exposed, under the central chimney and supported by suitable insulators on an iron spider lying on the floor of the oven. Its resistance when hot is about 17 ohms and the average current strength to maintain 120° C. is about 4.2 amperes., which means that the average power consumption is 300 watts. *R* is a recording thermometer, whose bulb is located horizontally beside the thermostat bulb. *B* is a low-form gas washing bottle, capacity 250 cc., which is filled three-quarters full with some good grade of transformer oil and serves as a tell-tale bottle to determine the rate of admission of air through the inlet tube, *H*. Air is passed through the oven at an approximate rate of 1.5 cu. ft. per hour, which is about equivalent to three or four bubbles per second when the internal diameter of the glass tube leading through the gas washing bottle is $\frac{3}{16}$ in. The revolving table inside of the oven is fitted with eight asbestos board seats, each of which holds a 600-cc. Pyrex beaker, Griffin form, approximately $3\frac{1}{4}$ in. in inside diameter and $4\frac{3}{4}$ in. high, containing 500 cc. of the sample to be tested.

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[1] General Electric Co., Pittsfield, Mass.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 448 (1923); Vol. 24, Part I, p. 638 (1924).

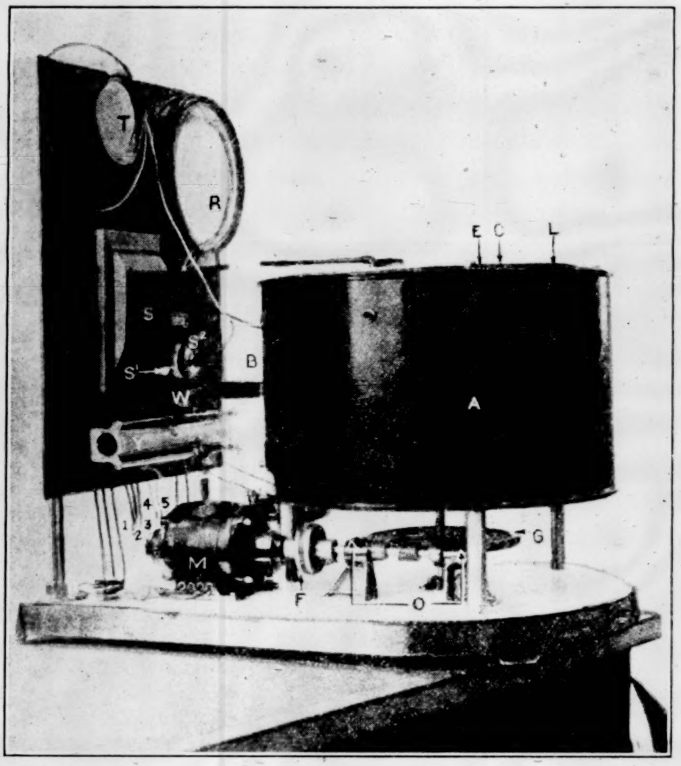

FIG. 1.—Apparatus Used in Life Test of Transformer Oils.

ADJUSTMENT OF APPARATUS

When starting a new machine for the first time, proceed as follows:

1. Fill all the spaces in the apparatus with samples of oil, (500 cc. of oil

in a 600-cc. beaker) and adjust the air flow through wash bottle *B* to about 1.5 cu. ft. per hour.

2. Start the motor which rotates the samples by closing switch *S*.

3. Move the thermostat pointer beyond the 140° C. mark, that is, off the scale, by screwing down the thumb screw on the side of thermostat *T*.

4. Turn on snap switch, S_2, of heater circuit (110 volt, D. C.) and move slide contacts *W* and *Z* of the rheostat *Y* to the center of rheostat and as close together as possible. Switch, S_1, must be open during this operation and kept open until time as noted under item No. 7.

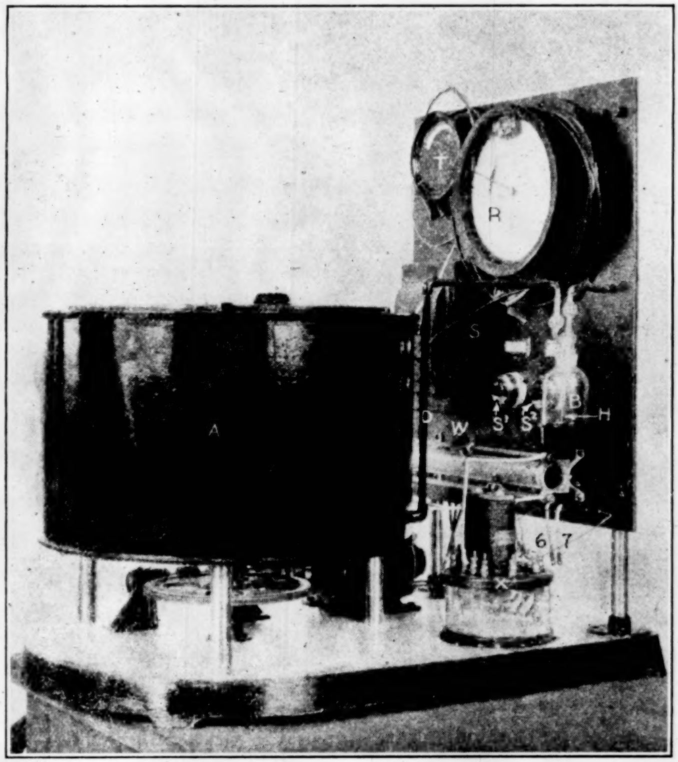

FIG. 2.—Apparatus Used in Life Test of Transformer Oils.

5. Move slide contacts, *W* and *Z*, keeping them close together, until recorder reads about 110° C. Several hours should be allowed to let the oven come up to full temperature.

6. Move the sliding contact *Z* controlling the relay *X*, to the right away from sliding contact *W*, until recorder reads 130° C.

7. Leaving the two sliding contacts as they now are, set the thermostat pointer on the 120° C. mark and snap on the relay switch, S_1.

8. Now stop the rotating motor and measure accurately the temperature of the oil samples by inserting a thermometer through sampling hole *C* in the cover and into the oil. Measure at least five samples, or beakers. Set the recorder so that it registers the temperature found with the thermometer. Start the motor again and move the pointer on the thermostat up or down as may be necessary to bring the oil samples precisely to 120° C.

NOTES

1. It is imperative to have the thermostat and recorder tubing, which enters at the side of the oven, insulated with felt so that the tubing will not touch the sides of the oven. Furthermore, this felt must extend at least 6 in. along the tube measuring from outside wall of the oven. There must not be any heat leakage from the oven through the tubing. The inlet holes should be well packed with felt around the tubing.

2. If the recorder gives a line showing rather large irregular dips, with comparatively long intervals, it is an indication that either the thermostat bearings or point is sticking due to friction or dirty contacts, etc. If the line shows large and regular dips, it is a sign that there is too much play in the bearings. The thermostat bearings and contacts must operate perfectly to give good results. There must be no friction and neither may there be any looseness or play. When the machine is operating properly, it will give a continuous line of closely set jags lying on the 120° C. mean, within a range of ±0.5° C.

3. The thermostat bulb and the recorder bulb must be placed in a horizontal position above the beakers in a plane just level with the top edge of the asbestos-covered central steel chimney. The air temperature here is at a maximum. They must not be placed directly over the chimney, where they would be heated mainly by radiation, but should be placed where they get the effect of the convection currents.

4. It is not necessary to have exactly 1.5 cu. ft. of air passing into the oven per hour. Actual tests have shown that this amount can vary from about 0.5 to 2.5 cu. ft. per hour without changing the results of the test.

PROCEDURE OF TEST

After the machine has once been adjusted, it should be capable of continuous operation with practically no further adjustments. The procedure of the test is as follows:

1. It is imperative to have the beakers absolutely clean. A very slight film of varnish from a previous test, even though the film is barely discernible when the beaker is held to the light, is enough to cause an error in the test, due to the accelerating catalytic action of the slight amount of oxidized oil film. Therefore, clean the beakers as follows:

Wash first with benzol, then with a cleaning solution of potassium bichromate and sulfuric acid. Then, after rinsing with water, scrub with scouring soap or detergent and water, rinse thoroughly with water and again rinse with cleaning solution. Rinse again thoroughly with water, finishing with distilled water and dry the beakers in a hot closet free from dust. Do not dry the beakers with towels.

2. Filter into the clean beakers through a good quality, dry filter paper, 500 cc. of the oil sample. Be careful not to contaminate the oil by measuring it with an unclean graduate or filtering through an unclean funnel. It is a good plan to have an etched mark on the side of the beaker indicating the 500-cc. capacity mark and then filter directly into the beaker from the sample can.

3. When the oven has been adjusted to the operating temperature, as noted under "Adjustment of Apparatus," the lid is removed, the eight beakers with their oil samples quickly put in place and the lid then replaced. The oven temperature will, of course, drop, but it should come up to temperature in about 1½ hours. The time at which the oil samples are placed in the oven is noted and considered as the beginning of the run, although the recorder will not show the test temperature for one to two hours, due to the cooling which takes place while putting the samples in the oven. As the oxidation at this low temperature (120° C.) is quite slow, several days at least will usually have elapsed before any sedimentation occurs, even with the poorest grades of oil. Once a day, the motor is stopped and the samples are examined by momentarily lifting a specimen of the oil in a clean 10-cc. pipette, which is inserted in the sampling hole, *C*. Before drawing up the sample, the oil should be stirred with the pipette. (See Note 8 below.) As soon as any sample is found to be near the sludging condition, 10 cc. of the oil are removed and placed in a 15-cc. centrifuge tube having a tapering end. From this time on, specimens are withdrawn daily from the oils which are suspected of sludging. The specimens, properly labeled with the date and number of the sample, are allowed to stand overnight and are centrifuged at 1800 r.p.m. for twenty minutes. The tubes are then withdrawn and examined. The number of days which elapse before the first trace of sludge appears in this specimen (see Note 12 below) is recorded as the time required to produce sludging in the sample from which it was removed. The time interval counted is the time from when the sample is placed in the oven until the time when the first trace of sludge is noted in a specimen removed from the sample. The time the specimen stands outside of the oven before centrifuging is not counted. All specimens must be examined within twenty-four hours after removal from the oven.

NOTES

5. An operator, after some experience with this method, will be able to judge fairly accurately from the darkening of the oil samples when they are approaching the sludging period. When starting with an unknown oil it may be necessary to remove specimens quite early and frequently and then repeat the run without removing specimens, allowing it to proceed closely to the time when sludging first would start. The point to be borne in mind, of course, is that the test will check more accurately with the minimum number of specimens removed before sludging begins. In case the first specimen withdrawn shows sediment, the tests must be repeated and earlier specimens taken.

6. Sometimes oil specimens after being removed from the oven and allowed to stand in the centrifuge tubes may deposit sludge two or three days after standing. It is important therefore that all specimens be observed *within* the 24-hour period after removal from the oven. Any results obtained after this 24-hour period should be disregarded. A good plan is to withdraw specimens just before the close of the day and examine them the first thing in the morning. Specimens should not be allowed to stand in direct sunlight, nor should they stand near steam pipes or radiators or in similar locations where they will be kept warmer than the prevailing room temperatures ranging from about 20 to 30° C.

7. It is also imperative, of course, that the samples are not contaminated during the run. Avoid inserting into the oil pipettes that are not absolutely clean and dry when taking samples. A clean dry pipette should be used for each sample.

8. Stir the oil with the pipette before taking the sample, but do not stir it by blowing through the pipette. It can be sufficiently stirred mechanically with a long stem pipette, inserted through the sampling hole.

9. It is good practice to check the recorder by taking daily checks of the oil temperatures with a thermometer inserted through the sampling hole. The oil should be kept at 120° C. The importance of keeping the oil at a uniform temperature of 120° C. can be judged from the fact that it has been proved that for one type of transformer oil, an increase of 8 to 9° C. doubles the rate of oxidation. If too many specimens have been removed from a sample, the temperature of the remaining oil may rise above 120° C. while the temperature in the fuller beakers may register 120° C. If this occurs, it is well to check the sample in a new run and start removing specimens at a later date. A safe rule to follow is to repeat the test when more than a total of 100 cc. of the specimen have been removed from the sample before the sludging point has been reached.

10. The total resistance of the rheostat should be so chosen that on the hottest day the minimum heating current (relay contacts open) will not raise the oven temperature quite to 120° C. Conversely, the sliding contacts should be adjusted to such a distance apart that when the oven is in operation the recorder chart shows not a smooth wavy line, but a line full of very closely set jags lying on the 120° C. mean.

11. It is, of course, not necessary to wait until all the samples in the oven have reached the sludging point before starting a new series of samples. As soon as one sample is sludged, it can be quickly removed and another sample quickly inserted. The slight drop of temperature in the oven, due to removing the cover for a short time, will be regained in less than an hour and the error introduced by this short-time drop, when compared to the total life of the oil, is negligible.

12. *Haze Point and Sludge Point.*—It will be noticed, on examining a centrifuged sample in front of a concentrated light, that it remains clear for the greater part of its life. About two or three days before it may be expected to sludge, a haze may develop, which can very plainly be seen with the concentrated light behind. This haze, of course, is incipient sludge—sludge in colloidal form. The oil at this point may be said to be saturated with the oxidized bodies produced during the run, which up to this point have gone into true solution and incidentally, greatly darkened the oil. After the saturation point is reached, the colloidal particles become more and more coarse as they become more and more numerous, until the sludging point is reached. This is defined as the point at which the first trace of deposit is seen at the bottom of the centrifuge tube in a sample which has been centrifuged after standing for not more than 24 hours after its removal from the oven.

DISCUSSION

[Mr. Farmer submitted on behalf of Committee D-9, revisions in the Tentative Methods of Testing Insulating Varnishes (Serial Designation: D 115 - 24 T).—Ed.]

Mr. Herschel. Mr. W. H. Herschel.[1]—Referring to the Tentative Methods of Testing Insulating Varnishes and the recommendation for determining viscosity, it is not clear to me whether the introduction of the Stormer instrument or the introduction of the MacMichael instrument is new. As I see the present situation, alternative methods for determining viscosity are given, but conversion factors between the two instruments are entirely lacking and are very likely impossible to obtain. I believe that this is laying the foundation for prolonged controversy over conversion factors between the two different methods of measuring viscosity.

Mr. Farmer. Mr. F. M. Farmer[2] (*Chairman of Committee D-9*).—I might say that both of these instruments have been in our tentative standard since its initial publication in 1921. The Stormer instrument is used to a very considerable extent in industry and we felt it was necessary to include it. We are not recommending standard tests—we are only recommending standard procedures and we are not saying which one should be used. When we get to specifications, then perhaps we will have to face that problem.

Mr. Herschel. Mr. Herschel.—I should like to ask whether it would not be possible to insert the provision that viscosity shall be expressed in absolute units and then mention the Stormer and the MacMichael as suitable instruments for the determination of viscosity. That would avoid this difficulty about conversion factors and it would simply be a question of accuracy in the determination of absolute viscosity. As it now stands we have viscosity expressed in absolute units by the MacMichael and viscosity expressed in another unit by the Stormer instrument. One man will use one instrument; another man will use the other type of instrument in another laboratory and then the controversy will begin. Both will be within their rights because they are using permissible instruments.

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[1] Associate Physicist, U. S. Bureau of Standards.

[2] Chief Engineer, Electrical Testing Laboratories, New York City.

Mr. Farmer. MR. FARMER.—The committee will be very glad to consider this suggestion. It will go into the records and will be referred to the subcommittee concerned.

[The revisions submitted by the committee were approved and the methods continued as tentative.—ED.]

REPORT OF COMMITTEE D-11
ON
RUBBER PRODUCTS

Two meetings have been held during the year in addition to the one during the present annual meeting: one on December 19, 1924, at New York, and one on March 26, 1925, at Philadelphia.

STANDARDS AND TENTATIVE STANDARDS

Of the standards and tentative standards of the Society, eleven standards and three tentative standards deal with rubber products. Committee D-11 has no recommendation to make with respect to the eleven standards. It recommends that the three tentative standards be continued in their present status.

NEW TENTATIVE STANDARDS

As indicated in its report of last year, the committee feels that there is little need for additional standards because the principal engineering applications for rubber products have already been reasonably well provided for. The principal activity of the committee is, and probably will continue to be, the development of performance tests referred to elsewhere in this report.

The committee has, however, been requested to prepare specifications for rubber disks used in alarm valves on sprinkler systems. While the money value of the total annual production of these disks is an extremely small percentage of the total of all rubber products used in engineering applications, the importance of quality is extremely great because the false operation of a sprinkler system valve may cause much damage.

A sub-committee, consisting of the rubber manufacturers concerned, the two large insurance organizations' laboratories represented on Committee D-11, a fire extinguisher manufacturer, a sprinkler manufacturer and others, has, after considerable preliminary study and experimentation, prepared specifications. While these have much narrower chemical limits and call for a harder disk than the first draft, as a result of tests, the sub-committee does not feel that it has yet had sufficient field experience with this final draft to justify submitting it as a tentative standard at this time. A considerable number of disks made in accordance with these latest requirements are however in service and are being periodically inspected, and it is the hope of the sub-committee that it will be prepared to make a definite recommendation next year. The evidence so far is that a very much better product will be obtained than heretofore and that the disks will prove satisfactory during a reasonably long life. In the meantime it is desired to publish the essential features of the specification as information. They are as follows:

1. *Material Covered.*—These specifications cover rubber disks for alarm valves in sprinkler systems. The service of these disks is such that they are wet on both faces at all times.

MANUFACTURE

2. (*a*) The disks shall be made of a properly vulcanized rubber compound that will meet all the requirements of these specifications. They shall have the two faces parallel and smooth and free from pitting, blisters, air checks and other surface defects.

(*b*) These disks shall be either molded or cut from sheet rubber. If cut from sheet stock, the circumference shall be concentric with the hole in the center and all cut edges shall be smooth.

CHEMICAL PROPERTIES AND TESTS

3. The compound from which the disks are made shall comply with the requirements in the following table:

Organic acetone extract, max	4.00	per cent
Chloroform extract, max	1.00	"
Alcholic KOH extract, max	2.00	"
Free sulfur, max	1.25	"
Total sulfur, max	4.75	"
Ash, min	57.00	"
Ash, max	65.00	"

4. (*a*) *Method of Analysis.*—The chemical analysis shall be made in accordance with the appropriate sections of Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1]

(*b*) The sulfur in barytes shall not be included in the allowable total sulfur. It is not the intent of these specifications to bar the use of carbon black but, when used, it shall be determined and considered as a mineral filler, and as such will be included in the allowable ash content.

PHYSICAL PROPERTIES AND TESTS

5. *Specific Gravity.*—The specific gravity shall not be less than 1.65 nor more than 1.80.

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[1] 1924 Book of A.S.T.M. Standards.

6. *Plastometer Readings.*—The sample disk shall be placed on a firm, hard, horizontal surface and a load of 1.0 kg. (2.2 lb.) applied through a spherical member 3.2 mm. (0.125 in.) in diameter. The indentation in millimeters one minute after the application of the load shall be called the plastometer reading, and such reading shall not be less than 30 mm. nor more than 40 mm.

7. *Number of Tests.*—At least three tests shall be made on each face of each disk tested and the average shall be taken as the plastometer reading The results of all tests, however, shall be within the specified limits.

8. The temperature of the disks at the time of making plastometer readings shall not be less than 15.5° C. (60° F.) nor more than 26.7° C. (80° F.) If the disks have been stored in a temperature outside of these limits, they shall be subjected to a temperature within these limits for not less than 30 minutes immediately preceding the test.

DIMENSIONS

9. The dimensions shall be as specified by the purchaser. They shall not vary more than $\pm\frac{1}{32}$ in. from those specified.

MARKING

10. Each disk shall be marked with the manufacturer's name, the month and year of manufacture and the letters "A.S.T.M." These letters shall be not less than $\frac{1}{4}$ in. in height and shall be recessed in the face of the disk so as not to prevent the disk from being in intimate contact with the clapper of the valve.

NUMBER OF SAMPLES TESTED

11. The number of samples to be selected for test shall be determined by the purchaser.

INSPECTION AND REJECTION

12. (*a*) The purchaser may make the inspection and tests in his own laboratory or elsewhere. Such inspection, however, shall be made at the expense of the purchaser.

(*b*) If the inspection and tests are made at the place of manufacture, the manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the disks are being furnished in accordance with these specifications. Such inspection shall be so conducted as not to interfere unnecessarily with the manufacturer's routine.

13. *Rejection.*—All valves failing to meet the requirements of these specifications shall be rejected.

MISCELLANEOUS

The committee has considered the matter of an additional specification for adhesive tape for general use for electrical purposes, that is, one which would call for a higher grade material. The committee is not satisfied, however, that the demand for such a tape, which would necessarily be a more expensive one, is sufficient to justify the preparation of a specification. The evidence appears to be that the existing Standard Specifications for Adhesive Tape (D 69 – 24) meet the requirements of the consumers of the great bulk of the total production.

As previously indicated, much attention is being given by the committee to the subject of performance tests. It has long been recognized that a performance type of test is much more satisfactory, not only because it determines directly rather than indirectly the most important characteristic of the product, namely, life, but it is less restrictive on development. The subject is a very large one and much research work must be done in developing satisfactory methods before the committee will be in position to make definite recommendations. But a great deal of research work is actively under way by a sub-committee under the chairmanship of Mr. C. R. Boggs and definite progress is being made, particularly in the development of the following four types of test which the committee believes are the most important and should therefore receive first consideration:

1. *Abrasion Test.*—This is a test which is being developed for general application to those rubber products where abrasion in service is the most important element in determining the life of the material, such as conveyor belting, automobile tires, rubber-covered air and water hose, rubber heels, etc.

2. *Accelerated Aging or Life Test.*—This test is applicable to all rubber products since all such materials suffer natural deterioration with time, whether in service or in storage. An accelerated test which will show, in a relatively short time, the rate of deterioration which would naturally take place over a period of months or years due only to exposure to normal atmospheric conditions, is obviously a very important one. Two such types of test have been developed, one the Geer test, which has been in use for a number of years, and the other, the Bierer-Davis oxidation test, which is a recent development. A number of laboratories represented on the committee are making comparative tests on various types of rubber compounds with these two methods in conjunction with specimens of the same samples subjected to natural aging. The committee feels that it will have during the next year ample direct experimental evidence to justify recommending a definite accelerated life testing procedure. The next step would be the establishment of limits for various products.

3. *Flexing Test for Belting.*—This test is obviously intended for specific application to rubber belting. The committee has developed a special machine for testing samples of belting relatively quickly under conditions which involve those deteriorating influences which are encountered in actual service. A series of

tests will be made in several laboratories with this machine. There will also be some opportunity to correlate the results of these tests with actual service experience. It is the expectation of the committee that it will be possible to definitely predict the approximate life of belts in service from this empirical test and that therefore limits can be determined for inclusion in the specification for rubber belting.

4. *Oil and Gasoline Test.*—This test is also a specific one for rubber hose to be used in conveying oil and gasoline. The sub-committee dealing with this matter has collected considerable information but is not yet prepared to make a definite recommendation.

This report has been submitted to letter ballot of the committee, which consists of 42 members, of whom 29 have voted affirmatively, none negatively, and 13 have refrained from voting.

Respectfully submitted on behalf of the committee,

F. M. FARMER,
Chairman.

E. H. GRAFTON,
Secretary.

REPORT OF COMMITTEE D-13
ON
TEXTILE MATERIALS

Committee D-13 on Textile Materials held two meetings during the past year. The fall meeting was held in Providence on November 14 and 15 immediately following the meeting of the National Cotton Manufacturers Association in Boston. The spring meeting was also held at Providence on March 6 and 7. Both meetings were well attended by members and guests.

Four new sub-committees were organized during the past year:

Sub-Committee XIII on Narrow Fabrics;
Sub-Committee XIV on Rope and Cordage;
Sub-Committee XV on Rayon (Artificial Silks);
Sub-Committee XVI on Asbestos Textiles.

Sub-Committee XIII divided its work into elastic and non-elastic narrow fabrics and confined its efforts for the present to the latter. This sub-committee has held several meetings and has done a great amount of work but is not yet prepared to submit a tentative standard.

Sub-Committee XIV on Rope and Cordage and Sub-Committee XV on Rayon (Artificial Silks) and Sub-Committee XVI on Asbestos Textiles have outlined the work to be undertaken during the coming year and in each case it was decided to first cover definitions and nomenclature and requirements for test methods and tolerances. The work of these three sub-committees will broaden the scope of the work of Committee D-13 by entering three branches of the textile industries heretofore untouched by the committee.

Committee D-13 submits to the Society three new tentative standards:

1. Tentative Specifications for Tolerances for Numbered Cotton Duck.[1]
2. Tentative Specifications for Tolerances and Test Methods for Knit Goods.[2]
3. Tentative Methods of Testing Grease Wool and Allied Fibers for Scoured Content.[3]

The first has been given very careful consideration and in the main has been formulated long enough to get any adverse reactions from try-out and seems to be quite satisfactory. The second also has been given careful consideration but has not been formulated long enough to have the reactions from a try-out that are desirable. The third has not received as much attention from the sub-committee as the first two and may be subject to a very considerable criticism and revision, but it seemed advisable to publish it in order to get a general reaction from the committee.

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[1] See p. 840.—Ed. [2] See p. 842.—Ed. [3] See p. 846.—Ed.

Committee D-13 recommends that five tentative standards be advanced to standard as follows:

1. Tolerances for Hose Ducks and Belt Ducks (D 181 – 24 T);[1]
2. Tolerances and Test Methods for Electrical Cotton Yarns (D 203 – 24 T)[1];
3. Imperfections and Tolerances for Square-Woven Tire Fabrics (D 122 – 23 T)[2];
4. Imperfections and Tolerances for Cord Tire Fabrics (D 179 – 23 T)[2];
5. Specifications and Tests for Osnaburg Cement Sacks (D 205 – 24 T).[1]

All five of these tentative standards have been long enough before the committee to warrant that they will be found satisfactory in general use both to producer and consumer.

The Tentative Specifications for Tolerances for Hose Ducks and Belt Ducks (D 181 – 24 T) and the Tentative Specifications for Tolerances and Test Methods for Electrical Cotton Yarns (D 203 – 24 T) are being recommended for advancement to standard in their present form with the exception of a slight change of wording in the definition for "Direction of Twist" appearing in the latter specifications, making the definition read as follows:

> "*Direction of Twist.*—In the case of yarn, the yarn has right-hand twist if, when it is held vertically, the spirals or twists are seen to incline upward in a right-hand direction, and has left-hand twist when the spirals or twists are seen to incline upward in a left-hand direction."

In the case of the other three tentative standards, however, certain revisions are recommended as follows:

Tentative Specifications for Imperfections and Tolerances for Square-Woven Tire Fabrics (D 122 – 23 T).—

Section 7.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, pp. 1063, 1071, 1079 (1924); also 1924 Book of A.S.T.M. Tentative Standards, pp. 653, 661, 669.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, pp. 890, 894 (1923); also 1924 Book of A.S.T.M. Tentative Standards, pp. 645, 649.

> "On a basis of [four] *two* major defects or [twenty] *ten* minor defects, or any combination of these, being permissible in a roll of 100 sq. yd., the scoring shall be as follows:
>
> Deduct [25] *50* from 100 for each major defect in 100 sq. yd.
> Deduct [5] *10* from 100 for each minor defect in 100 sq. yd.
> Add the remainders, which sum shall be the score.

Section 9.—Change the examples given in Section 9 to agree with the change in Section 7 given above.

Section 15.—Change the permissible tolerances on bow from 0.75 per cent to 0.65 per cent.

Section 16.—Change the allowable variation on off-square from 3 per cent to 2 per cent.

Section 17.—Change the permissible variation on thickness from 5 per cent to 4 per cent in the case of combed staple and from 7.5 per cent to 5.5 per cent in the case of carded staple.

Section 19.—Add a new Section 19 on twist to read as follows:

> "19. *Twist.*—The average ply twist found by test shall not vary more than 5 per cent over or under the specified twist."

Some few changes in the definitions appearing in these specifications are being made at the suggestion of Committee E-8 on Nomenclature and Definitions, but these changes are all matters of form, the substance being not involved.

Tentative Specifications for Imperfections and Tolerances for Cord Tire Fabrics (D 179 – 23 T).—

Section 7.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> "On a basis of [four] *two* major defects or [twenty] *ten* minor defects, or any combination of these, being permissible in a roll of 100 sq. yd., the scoring shall be as follows:
>
> Deduct [25] *50* from 100 for each major defect in 100 sq. yd.
> Deduct [5] *10* from 100 for each minor defect in 100 sq. yd.
> Add the remainders, which sum shall be the score."

Section 9.—Change the examples given in Section 9 to agree with the change in Section 7 given above.

Section 15.—Change the permissible variation on thickness of cord made of carded staple from 5 per cent to 4 per cent.

Some few changes in the definitions appearing in these specifications are being made at the suggestion of Committee E-8, but these changes are all matters of form, the substance being not involved.

Tentative Specifications and Tests for Osnaburg Cement Sacks (D 205 - 24 T).—

Section 5.—Change the present Section 5 to Section 5 (*a*) and add a new Paragraph (*b*) to read as follows:

"The weight shall be determined under prevailing atmospheric conditions except in the settlement of disputes, in which case the test for weight shall be made upon material having a normal moisture content obtained by exposure for at least 4 hours to an atmospheric condition of 65-per-cent relative humidity at a temperature of 21° C. (70° F.)."

Section 7.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

"(a) The *average* strength in both the warp and filling shall be *not less than that specified by the purchaser.* [75 lb. with a tolerance of not more than 5 lb. under the desired tensile strength.]

"(b) *The tensile strength shall be obtained on an approved type of inclination balance machine, motor driven.* The tensile strength shall be determined under prevailing atmospheric conditions except in the settlement of disputes, in which case the test for tensile strength shall be made upon material having a normal moisture content obtained by exposure for at least 4 hours to an atmospheric condition of 65-per-cent-relative humidity at a temperature of 21° C. (70° F.)."

The Tentative Definitions of Terms Relating to Textile Materials (D 123 - 22 T) and the Tentative Methods of Testing Cotton Fibers (D 152 - 22 T) are recommended to remain another year as tentative without change. The Tentative Specifications for Tolerances and Test Methods for Cotton Yarns, Single and Plied (D 180 - 24 T) and the Tentative Specifications for Tolerances and Test Methods for Cotton Sewing Threads (D 204 - 24 T) are recommended to be revised as follows and continued as tentative in their revised form:

Tentative Specifications for Tolerances and Test Methods for Cotton Yarns, Single and Plied (D 180 - 24 T).[1]—

Section 4.—Change the definition for the term "direction of twist" to read as follows:

"*Direction of Twist.*—In the case of yarn, the yarn has right-hand twist if, when it is held vertically, the spirals or twists are seen to incline upward in a right-hand direction, and has left-hand twist if the spirals or twists are seen to incline upward in a left-hand direction."

Section 6.—After the present second sentence, add a new sentence to read as follows:

"The drums of the testing machine shall consist of cylindrical spools not less than one inch in diameter and not less than one inch in width, so supported that at least one shall turn freely upon its axis."

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 1066 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 656.

Section 7.—Add a new Section 7 to read as follows, renumbering the subsequent sections accordingly:

"7. *Strength Yarn on Beams (Skein Test for Yarn on Beams).*—Yarn received on beams shall be given the skein test and skeins shall be prepared in the following manner. Place the beam containing the yarn to be tested on two bearings sufficiently high from the floor so that the beam can be turned easily. Directly in front of the beam and at least 15 ft. from the beam, a small table shall be placed, the top of which will be approximately the same height as the top of the beam of yarn. On this table a standard yarn reel shall be placed directly in front of the beam. A small crank arm shall be attached to one end of the beam shaft. Four ends of yarn, taken from the beam, shall be attached to the yarn reel in the regular manner, such as is done when testing yarn from bobbins. One operative shall turn the beam slowly in the proper direction so that the yarn will unwind, and the second operative shall turn the reel fast enough to take up the yarn as it comes off the beam. This operation shall be continued until 120 yd. or the desired length for testing has been taken from the beam. The four skeins shall then be taken from the reel and tested in the regular manner. If it should be necessary to test more ends, the same method of testing shall be followed.

"NOTE.—In using these beams after these ends have been taken off, a set of spools containing the same kind of yarn as is on the beam can be placed in behind the beam on a small creel, to fill out the number of ends that have been used for testing. When the ends come up on the beams that have been used for testing, the auxiliary spools in back of the beams can be broken out."

Section 7.—Omit from the fourth to the last sentences reading as follows:

"This test shall be used for yarns received on beams, etc. To prepare specimens for test, at least 60 ends, 48 in. in length, shall be unwound from the beam. The bundle shall be held at one end to allow ends to twist upon themselves. A knot shall be tied in each end and the bundles allowed to condition as specified in Section 7. After conditioning, one end of the bundle shall be clamped in the head end jaw of any twist counter, and the other end shall be held by any suitable means, applying approximately the same tension as is used in preparing skeins on reels. The bundle shall be untwisted until strands are parallel, 60 ends counted off and cut from the bundle to a measure of 1 yd. in length. Knots shall again be tied in each end and the size of the 60 yd. determined in accordance with Section 13. After the size has been determined the strands shall again be made parallel, removed one by one, and broken on a single strand tester. The average of 20 breaks shall be reported as the average strength."

Tentative Specifications for Tolerances and Test Methods for Cotton Sewing Threads (D 204 - 24 T).[1]—

Section 4.—Change the definition for the term "direction of twist" to read as follows:

"*Direction of Twist.*—In the case of sewing thread, the thread has right-hand twist if, when it is held vertically, the spirals or twists are seen to incline upward in a left-hand direction, and has left-hand twist if the spirals or twists are seen to incline upward in a right-hand direction."

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 1076 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 666.

Section 6.—Change to read as follows by the addition to the italicized words and the omission of those in brackets:

"In addition to meeting the foregoing requirements, sewing thread shall be subjected to a sewing machine test under actual factory conditions [and in the event that any thread causes more than a reasonable number of stoppages it shall be subject to rejection]. *In the event of any thread apparently failing to operate properly, it shall be judged by comparison on any given machine under identical conditions, with thread from a lot known to have previously operated satisfactorily.*"

Section 7.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

"All threads shall be tested by the single strand test method. A single strand tester of proper capacity with jaws set 10 in. between grips and having a speed of pulling jaw of 12 in. per minute shall be used. The average of [four] *ten* breaks from each of [ten] *five* spools or packages shall be the average strength. Any test during which the thread breaks at the jaw due to pinching or cutting shall not be included in the average."

Section 9.—Add a new Section 9 under the central heading "Length of Package," to read as follows, renumbering the subsequent sections accordingly:

"(*a*) (*Preferred Method*).—The length of package of sewing thread shall be determined by multiplying the number of yards per pound, obtained as outlined in Section 8, by the net weight in pounds of the thread in package to be tested.

"(*b*) (*Alternate Method*).—The length of package of sewing thread shall be determined by measuring the entire length over a reel having a $1\frac{1}{2}$ yd. perimeter, taking three full turns around the reel and delivering thread to a take-up drum. The yardage shall be noted on reel dial.

"(*c*) (*Alternate Method*).—The length of package of sewing thread shall be determined by measuring the thread in the form of skeins prepared on a reel having a $1\frac{1}{2}$ yd. perimeter. In no case shall the yardage of these skeins exceed that specified in Table II. The total yardage of skeins thus prepared from a package constitutes its length.

TABLE II

EQUIVALENT SINGLE SIZE	LENGTH OF SKEIN, YD.
Finer than 12's	240
6's to 12's, inclusive	120
Coarser than 6's	60

The Tentative Specifications for Textile Testing Machines (D 76 – 22 T)[1] have been completely rewritten and are appended hereto in their proposed revised form.[2]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 971 (1922); also 1924 Book of A.S.T.M. Tentative Standards, p. 644.

[2] See p. 829.—ED.

The recommendations contained in this report have been submitted to letter ballot of the committee, consisting of 118 members, with the following results:

Items	Affirmative	Negative	Not Voting
I. TENTATIVE STANDARDS ADVANCED TO STANDARD			
Tolerances for Hose Ducks and Belt Ducks (D 181 – 24 T)	44	0	24
Tolerances and Test Methods for Electrical Cotton Yarns (D 203 – 24 T)	35	3	30
Imperfections and Tolerances for Square-Woven Tire Fabrics (D 122 – 23 T)	42	2	25
Imperfections and Tolerances for Cord Tire Fabrics (D 179 – 23 T)	42	3	24
Specifications and Methods of Test for Osnaburg Cement Sacks (D 205 – 24 T)	41	3	25
II. REVISIONS OF TENTATIVE STANDARDS			
Tolerances and Test Methods for Cotton Yarns (D 180 – 24 T)	50	2	19
Tolerances and Test Methods for Cotton Sewing Threads (D 204 – 24 T)	43	0	26
Tentative Specifications for Textile Testing Machines (D 76 – 22 T)	55	5	11
III. NEW TENTATIVE STANDARDS			
Tentative Specifications for Tolerances for Numbered Duck	44	0	24
Tentative Specifications for Tolerances and Test Methods for Knit Goods	35	2	32
Tentative Methods of Testing Grease Wool and Allied Fibers for Scoured Content	33	1	34

The Special Sub-Committee on Membership has been very active in extending the membership of Committee D-13 and has added 25 new members during the past year. The sub-committee has been requested to continue this service for another year, which it has agreed to do.

A brief statement of the progress and extension of the work of Committee D-13 since its inception in March, 1915, will be of value in showing the direction and scope of its work.

The first report of Committee D-13, in 1915, stated that the committee met on April 10 (1915) and "determined to limit its activities for the present to the consideration of tests and specifications of bags and bagging materials and tire fabrics." This resulted in Tentative Tests for Automobile Fabrics (D 31 – 16 T); Tentative Tests for Cotton Fabrics for Use in Hose, Belting and Similar Articles (D 32 – 16 T); Tentative Tests for Cotton Fabrics for Use in Bags and Bagging Material (D 33 – 16 T), and Tentative General Methods for Testing Cotton Fabrics (D 39 – 16 T). Only the last one has been advanced to standard, this recommendation being made in 1920. This was revised in 1923 by emendations and several additions and is published in the 1924 Book of A.S.T.M. Standards under the Serial Designation D 39 – 24.

This does not indicate indifference or lack of work on the part of this committee, but that there was a dearth of analyzed data and varied opinions due to individual and different methods of testing and a lack of publicity. The committee early saw the necessity for analyzing the tests and results of different individuals and laboratories in order to bring order out of the chaos. This has resulted in this one standard which is the foundation for methods of testing of textile fabrics whether of cotton or of other textile fibers.

We may reasonably expect rapid progress in bringing out textile standards of every kind now that a secure foundation for and awakened interest in testing textile fabrics and yarns has been brought about as indicated by the new sub-committees, the new tentative standards and the new standards covered in this report. These standards and tentative standards are the outcome of active and unprejudiced cooperation of all interested parties—producers, consumers, and technical laboratories including government bureaus and may confidently be expected to be put into common use expeditiously.

This report has been submitted to letter ballot of the committee, which consists of 131 members, of whom 66 have voted affirmatively, none negatively, and 65 have refrained from voting.

Respectfully submitted on behalf of the committee,

W. F. EDWARDS,
Chairman.

K. B. COOK,
Secretary.

EDITORIAL NOTE

The Tentative Specifications for Tolerances for Hose Ducks and Belt Ducks; for Tolerances and Test Methods for Electrical Cotton Yarns; for Imperfections and Tolerances for Square-Woven Tire Fabrics; and for Imperfections and Tolerances for Cord Tire Fabrics; and the Tentative Specifications and Methods of Test for Osnaburg Cement Sacks were approved at the annual meeting and subsequently adopted by letter ballot of the Society on August 28, 1925, and appear in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

The proposed revisions of the Tentative Specifications for Tolerances and Test Methods for Cotton Yarns, Single and Plied; for Tolerances and Test Methods for Cotton Sewing Threads; and for Textile Testing Machines were accepted. The tentative specifications as thus revised appear on pages **831**, 837 and **829**, respectively.

The proposed new Specifications for Tolerances for Numbered Cotton Duck; for Tolerances and Test Methods for Knit Goods; and the proposed Methods of Testing Grease Wool and Allied Fibers for Scoured Content were accepted for publication as tentative and appear on pages **840**, **842**, and **846**.

REPORT OF COMMITTEE D-14

ON

SCREEN WIRE CLOTH

During the past year Committee D-14 has held three meetings, at Atlantic City in June, in Washington in October, and a March meeting in Philadelphia, the latter two being in conjunction with the joint metal committees meetings.

The activities of the committee have in this time been concentrated on long-time exposure tests of representative non-ferrous screen wire materials. These tests are being undertaken under the direction of Sub-Committee IV on Testing, but are receiving the support of the entire committee in the furnishing of materials and facilities.

The purpose of these tests is to determine the relative life of the higher grade screen wire cloths when exposed to various atmospheric agencies, such as corrosion and wind, as they exist in different localities and climates, so that purchasers of such material may make an economic selection best suited to their particular conditions.

It is realized that such tests will of necessity cover a space of several years, and hence considerable time and thought has been given before starting them, in order to reduce the number of variables so far as possible and ultimately secure the most valuable data without the necessity of repetition. Some of the variables that are encountered may be listed as follows:

I. Screen Wire Cloth:
  - (*a*) Composition;
  - (*b*) Mesh;
  - (*c*) Size of wires;
  - (*d*) Protective coating on wires.

II. Frame:
  - (*a*) Material of frame;
  - (*b*) Size of frame;
  - (*c*) Tension of cloth in frame;
  - (*d*) Possibility of contaminating corrosive products from frame.

III. Exposure Location:

- (*a*) Composition of atmosphere;
- (*b*) Temperature;
- (*c*) Relation of exposure to prevailing winds and sun's rays;
- (*d*) Interference of test screens with each other.

How these various factors were disposed of will be apparent from the description of the tests. Seven different grades of wire have been used in weaving the cloth for the test pieces. Table I shows the composition of these materials as determined by analysis[1] of a sample from the finished cloth. The 90 copper - 10 zinc alloy and the unalloyed copper correspond respectively to Class A and Class B material of the Tentative Specifications for Non-Ferrous Insect Screen Cloth (Serial Designation: B 50 - 23 T) of the American Society for Testing Materials.[2] The other materials are all commercially obtainable under various trade names. All of the cloth was woven 30 in. wide in accordance with the above specifications in 16-mesh standard. No coating or lacquer was applied to any of the screen wire cloth.

TABLE I.—COMPOSITION OF MATERIALS USED IN EXPOSURE TESTS OF SCREEN-WIRE CLOTH.

Material	Copper, per cent	Zinc, per cent	Nickel, per cent	Tin, per cent	Iron, per cent	Lead, per cent	Manganese, per cent	Silicon, per cent	Aluminum, per cent
90 Copper, 10 Zinc..........	90.7	9.05[a]	not detected	0.23	less than 0.02	not detected	...	....	....
80 Copper, 20 Zinc..........	80.1	19.88[a]	not detected	....	less than 0.02	not detected	...	....	...
75 Copper, 20 Nickel, 5 Zinc	73.9	5.4	19.8	....	0.3	....	0.5	0.06	...
70 Nickel, 30 Copper (approx.)	26.5	....	68.3	....	2.4	....	2.7	....	...
Unalloyed Copper..........	99.94	not detected	not detected	less than 0.01	....	not detected	...	....	...
98 Copper, 2 Tin..........	98.3	not detected	not detected	1.8	less than 0.01	not detected	...	not detected	...
95 Copper, 5 Aluminum....	95.2	not detected	not detected	0.08	....	not detected	...	....	4.8

[a] By difference.

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[1] These analyses were made by the non-ferrous laboratory of the Bureau of Standards.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 591 (1923); also 1924 Book of A.S.T.M. Tentative Standards, p. 107.

Three types of frames are being used for supporting the test specimens. Two of these are typical of commercial screen sizes, being 30 by 36 in., one of wood and the other of copper. In these frames the cloth is stretched uniformly tight as in commercial practice. For the third type, a 12 by 12-in. (inside dimension) wooden frame is used in which the small test sample is loosely held by wedge pins, thus avoiding the use of any tension in the cloth. Both types of wooden frames are painted for the preservation of the wood during the long exposure anticipated. Care has been taken to use a paint that will not influence the corrosion of the screen wires, particularly freedom from sulfides and sulfates being essential. For this purpose a paint using only basic carbonate white lead in oil, linseed oil, painters' drier and turpentine was specified.

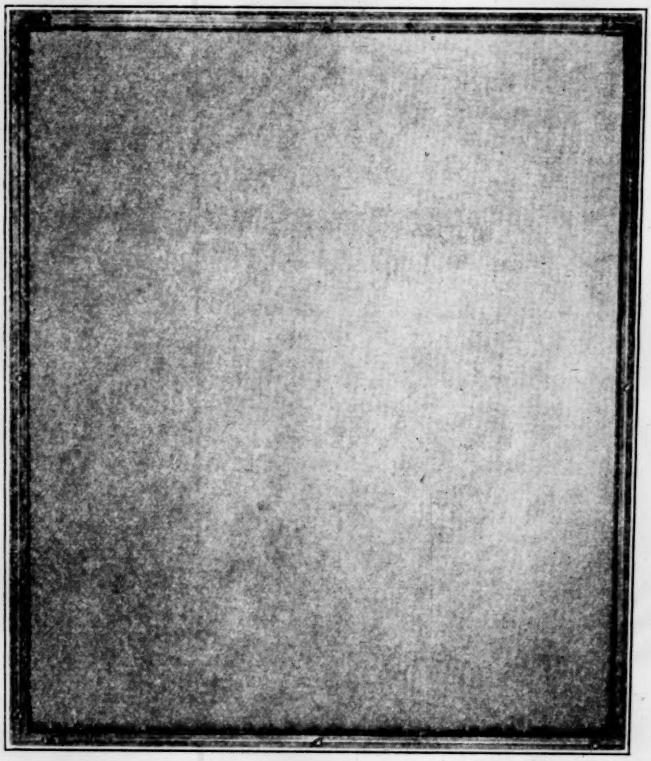

FIG. 1.—Screen Mounted on 30 by 36-in. Metal Frame.

The following is the analytical report on the paint used:[1]

Color	White
Odor	Turpentine
Consistency	Painting
Weight per gallon	17.1 lb.
Pigment, per cent by weight	64.3

Vehicle:

Volatile matter, per cent by weight, 6.0 = 16.8 per cent by weight of vehicle.

Non-volatile matter, per cent by weight, 29.7 = 83.2 per cent by weight of vehicle.

Nature of pigment: Basic carbonate white lead; no sulfates or sulfides.

The sample was a straight white lead paint.

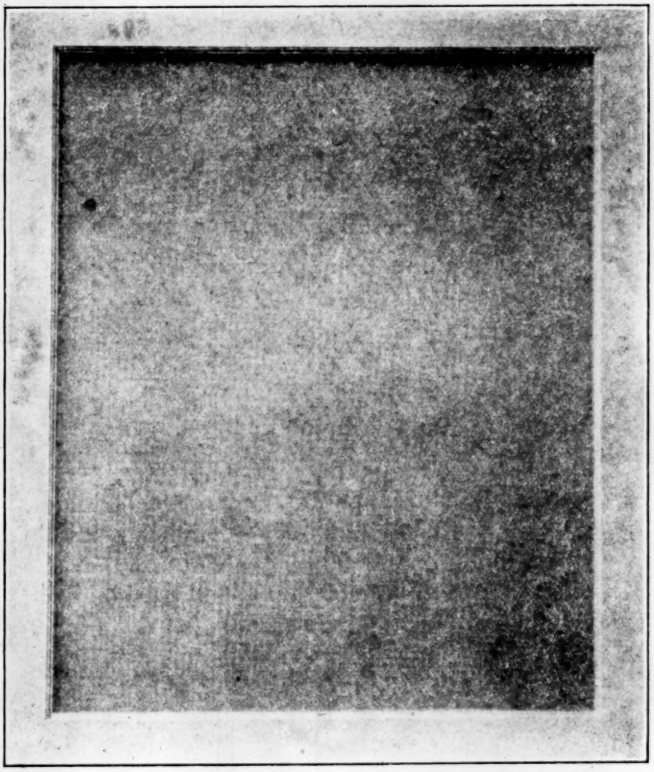

FIG. 2.—Screen Mounted on 30 by 36-in. Wooden Frame.

Figs. 1, 2 and 3 show photographs of the completed frames. Through the courtesy of several Government departments four exposure locations have been obtained which represent different corrosive conditions. These are at the Bureau of Mines at Pittsburgh, Pa., where there is a typical bad manufacturing atmosphere; at the Portsmouth, Va., Light House, which represents a normal sea coast atmosphere; at the Panama Canal, where the atmosphere is that of a tropical sea coast, and at the Bureau of Standards, Washington, D. C., representing a normal inland atmosphere.

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[1] Analysis furnished by Bureau of Standards.

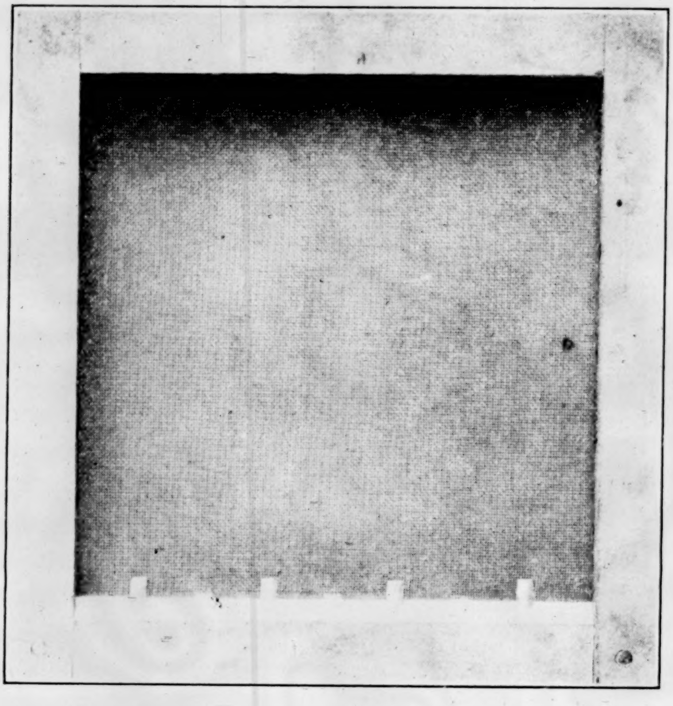

FIG. 3.—Screen Mounted on 12 by 12-in. Wooden Frame.

At each of these points there is one panel of each material in the three types of frames, making twenty-one panels at every test location, or a total of eighty-four test panels under observation. All frames are mounted with the shoot or straight wires in a horizontal position and are spaced at least one-half the width of the screen apart in order to relieve wind pressure. Screens are not placed above or in front of one another so as to avoid as far as possible the effect of the corrosive products of one screen upon another. Figs. 4 and 5 show general views of the exposure tests in place at the Bureau of Standards.

Sub-Committee IV is also preparing to undertake laboratory tests of the same materials which are being used in the long-time atmospheric corrosion tests. About 100 specimens, each 2 in. square, have been prepared for accelerated corrosion tests including (1) salt spray, (2) interrupted repeated immersion in (*a*) water, (*b*) dilute sulfuric acid, (*c*) salt solution. Tension tests and Erichsen cupping tests will also be made on the wire cloth.

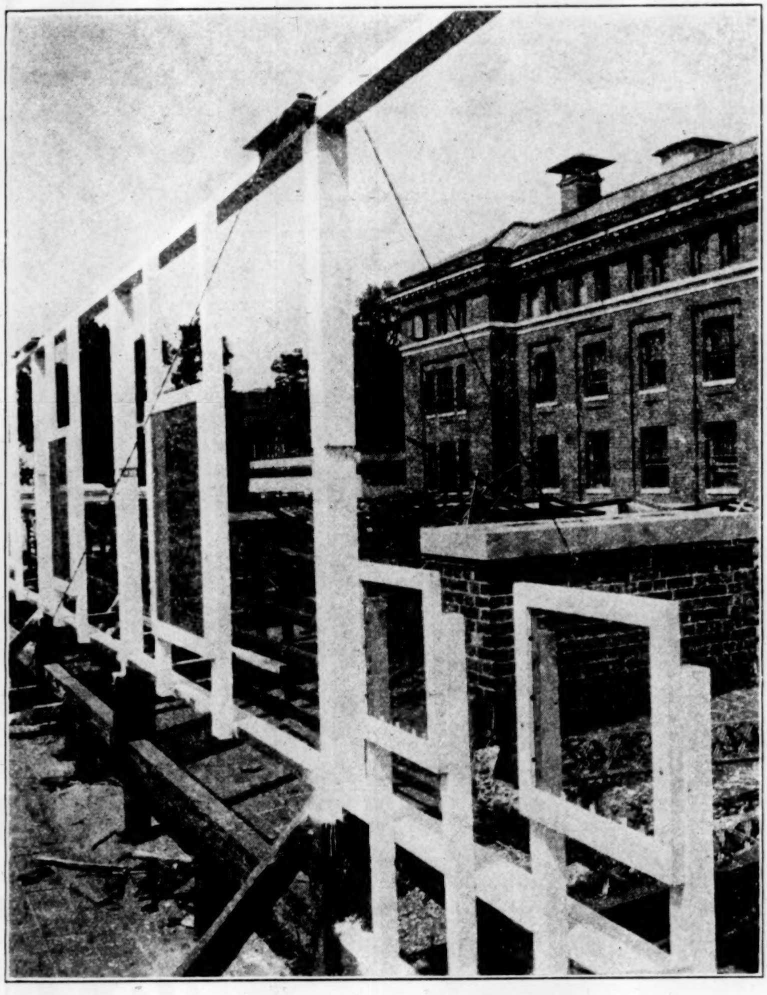

FIG. 4.—Shows Mounting of Screens in Wooden Frames.

Committee D-14 recommends that in the Tentative Specifications for Non-Ferrous Insect Screen Cloth (B 50 – 23 T), Section 12 be amended by making the section as it now stands Paragraph (*a*) and adding a new Paragraph (*b*) to read as follows:

"(*b*) Well-made splices in individual wires are permitted at any point in the screen cloth."

With this revision, Committee D-14 recommends that these specifications be continued as tentative. The result of the letter ballot of the committee on this recommendation is as follows: Affirmative, 11; negative, 0; not voting, 7.

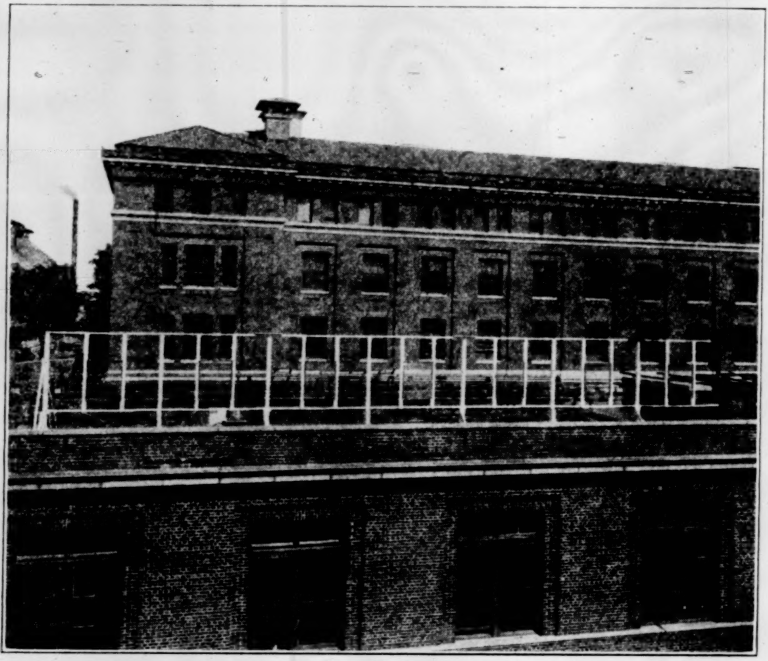

FIG. 5.—Shows Mounting of Screens in Metal Frames.

This report has been submitted to letter ballot of the committee, which consists of 18 members, of whom 13 have voted affirmatively, none negatively, and 5 have refrained from voting.

Respectfully submitted on behalf of the committee,

R. W. WOODWARD,
Chairman.

W. H. BASSETT,
Secretary.

EDITORIAL NOTE

The proposed revision of the Tentative Specifications for Non-Ferrous Insect Screen Cloth was accepted. The tentative specifications as revised appear on page 587.

REPORT OF COMMITTEE D-15
ON
THERMOMETERS

The committee has arrived at a standard form for thermometer specifications and a large proportion of the Society's thermometer specifications have now been put in this standard form. Among the earliest specifications to be put in the standard form are those for three thermometers for general use. Tentative Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −20 to +150° C., 0 to +300° F. (D 182 – 23 T),[1] Tentative Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +300° C., +20 to +580° F. (D 183 – 23 T),[1] and the Tentative Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +400° C., +20 to +760° F. (D 184 – 23 T).[1] The committee recommends that these be now advanced to standard with the addition of the following note:

"NOTE.—For the purpose of interpreting these specifications the following definitions apply:

"The total length is the over-all length of the finished instrument.

"The diameter is that measured with a ring gage.

"The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

"The top of the thermometer is the top of the finished instrument."

The present Method of Test for Cloud and Pour Points of Petroleum Products (D 97 – 23 T) provides for the use of a mercury thermometer, thus limiting the test to temperatures above −38° F. At the request of Committee D-2 on Petroleum Products and Lubricants, specifications for a low cloud and pour test thermometer, graduated to −60° C. or −70° F. were prepared and submitted to Committee D-2 as appended hereto.

In the use of the Saybolt viscosimeter, there have been many instances of damage to the instrument resulting from the end of the thermometer bulb coming in contact with the upper end of the capillary tube, resulting in minute deformations of the metal and resulting changes in the constants of the instrument. In order to provide means for avoiding such occurrences the committee has recommended that the stem of the thermometer be made with an enlarged portion, which may be used as a stop.

The committee has made some tests with the turpentine distillation thermometers, but proposes to make additional tests before making final recommendations.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, pp. 905–910 (1923); also 1924 Book of A.S.T.M. Tentative Standards, pp. 677–682.

In testing thermometers, a standard thermometer which can be used to a higher order of accuracy than the thermometers under test has usually been preferred. In such tests, the standard and the thermometers under test, to the extent that they differ in design, will not be affected to the same extent by the influences to which they are subject and such tests must therefore be made with great care and involve considerable calculation. Ultimately all tests must, of course, be referred to the reference standard. The committee wishes to call attention, however, to the advantages resulting from making routine tests against standards identical with the product tested, as tending toward the simplicity in procedure and uniformity in results.

The proposed specifications and the advancement to standard of the three tentative standards mentioned have been submitted to letter ballot of the committee with the following results:

Items	Affirmative	Negative	Not Voting
I. PROPOSED TENTATIVE SPECIFICATIONS			
1. A.S.T.M. Low Cloud and Pour Test Thermometer, −60 to +20° C. or −70 to +70° F.	8	1	2
II. TENTATIVE STANDARDS ADVANCED TO STANDARD			
2. Tentative Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −20 to +150° C., 0 to +300° F. (D 182 - 23 T)	10	0	1
3. Tentative Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +300° C., +20 to +580° F. (D 183 - 23 T)	10	0	1
4. Tentative Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +400° C., +20 to +760° F. (D 184 - 23 T)	10	0	1

This report has been submitted to letter ballot of the committee, which consists of 11 members, of whom 9 have voted affirmatively, none negatively, and 2 have refrained from voting.

Respectfully submitted on behalf of the committee,

W. H. FULWEILER,
Chairman.

E. F. MUELLER,
Secretary.

EDITORIAL NOTE

The proposed specifications for A.S.T.M. Low Cloud and Pour Test Thermometer, −60 to +20° C., −70 to +70° F., were accepted by Committee D-2 and incorporated in the Tentative Method of Test for Cloud and Pour Points of Petroleum Products.

The Tentative Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −20 to +150° C., 0 to +300° F.; for A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +300° C., +30 to +580° F., and for A.S.T.M. Partial Immersion Thermometer for General Use, −5 to +400° C., +20 to +760 F., were approved at the annual meeting and subsequently adopted by letter ballot of the Society on August 28, 1925, and appear in the supplementary pamphlet of A.S.T.M. Standards Adopted in 1925.

PROPOSED SPECIFICATIONS
FOR
A.S.T.M. LOW-CLOUD AND POUR TEST THERMOMETERS
−60 TO +20° C., −70 TO +70° F.

1. These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −60 to +20° C. or −70 to +70° F.

2. The purpose of these specifications is to provide a special low range thermometer suitable for use in cloud and pour tests.

3. The thermometer shall conform to the following requirements.

Type: Etched stem, glass.

Liquid: Toluene or other suitable liquid colored red with a permanent dye.

Range and Sub-division: −60 to +20° C. in 1° C. or −70 to +70° F. in 2° F.

Total Length: 227 to 231 mm. (8.92 to 9.08 in.).

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter 7.0 to 8.0 mm. (0.28 to 0.31 in.).

Bulb: Corning normal or equally suitable thermometric glass.
Length, 8.0 to 9.5 mm. (0.31 to 0.37 in.).
Diameter, 5.5 to 6.5 mm. (0.22 to 0.26 in.).

Distance to −57° C. or −70° F. Line from Bottom of Bulb: 120 to 130 mm. (4.73 to 5.12 in.).

Distance to +20° C. or +68° F. Line From Top of Thermometer: 35 to 45 mm. (1.38 to 1.77 in.).

Expansion Chamber: To permit heating to +60° C. or +140° F.

Filling Above Liquid: Gas under pressure.

Top Finish: Plain.

Graduation: All lines, figures and letters clear cut and distinct, lines at multiples of 5° C. or 10° F. to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

Immersion: 108 mm. (4¼ in.). The words "108-mm. immersion" on Centigrade thermometers, or "4¼-in. immersion" on Fahrenheit thermometers and a line around the stem 108 mm. (4.25 in.) above the bottom of the bulb, shall be etched on the thermometer.

Special Marking: "A.S.T.M. Low Cloud and Pour" a serial number and the manufacturer's name or trade mark shall be etched on the thermometer.

Scale Error: The error at any point of the scale, when the thermometer is standardized as provided below, shall not exceed 1° C. or 2° F.

Standardization: The thermometer shall be standardized at the ice point and at intervals of approximately 20° C. or 35° F. for 108-mm. or 4¼-in. immersion, and for an average temperature of 21° C. or 70° F. for the emergent liquid column.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking "A.S.T.M. Low Cloud and Pour, −60 to +20° C." or "A.S.T.M. Low Cloud and Pour, −70 to +70° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications, the following definitions apply.

The total length is the overall length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

REPORT OF COMMITTEE D-16
ON
SLATE

Committee D-16 since its organization on January 21, 1924, has held three meetings.

The limited membership of this committee has prevented a satisfactory organization of sub-committees but the various lines of endeavor have been undertaken mainly by individuals having the proper facilities at hand. Considerable attention has been devoted to the more common laboratory tests for determining the physical properties of the material.

A comprehensive series of tests has been made at the U. S. Bureau of Standards in this connection on the absorption of slate with a view of adapting a procedure to the special properties of this material. One point developed in this study is the fact that the most satisfactory shape of specimen is a thin slab rather than a cubical or cylindrical specimen, such as is used for this test on other types of natural stone. Due to the small amount of water absorbed it is necessary to employ more precision in weighing and surface drying before the final weighing than is the case for more porous materials. Also the natural cleavage of the material must be taken into consideration and the surface of the specimen should be rubbed to a smooth finish. All of these points have been given careful consideration both in the research on the subject and through discussion at the meetings at which the subject was viewed in the light of practical experience and actual service conditions. The committee feels that the procedure which it has adopted for the absorption test and is recommending as a tentative standard[1] will prove of value in establishing more uniform and reliable data on this important property.

Considerable attention has been given to methods of flexure tests in the study and discussion of data already available on the subject. While it may be argued that slate in actual use is seldom required to resist stresses, still it is important to have a ready means of comparing the strength and elasticity of slates from different deposits in order to gain a knowledge of their relative qualities. On account of the natural characteristics of slate this is most readily obtained by means of the flexure test. Methods of procedure for strength and elasticity tests are recommended[1] which employ the transverse bending of small slabs. It seems unfortunate that different shapes of specimens must be recommended for determining the strength and elasticity values, since this requires separate tests when the two properties are desired. Different shapes are recommended due to the fact that thicker specimens utilized in the strength tests can readily be cut from stock slabs of slate without appreciable waste of material. This seems to be justified since the elasticity of slate is not so often determined. For the latter determination a thin slab is recommended in order to obtain larger deflection measurements and thus avoid the use of the more precise measuring instruments. In order to keep the weight measurements correspondingly large, a greater width is recommended. In both cases, the committee has considered the fact that some laboratories or interests may desire to employ the tests where only light testing equipment is available. With this point in view, procedures are recommended which can be used on machines of a capacity of 1000 lb. Some attention has been given to adapting simple and inexpensive equipment for use in these tests.

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[1] See p. 744.—Ed.

The hardness of slate is of interest to the producer in that this property is an important factor in production costs, and to the consumer because it bears a relation to the service the material will give under certain conditions of use. Hence, any method of determining the hardness should give results which bear some definite relation to working hardness and abrasive hardness.

A very praiseworthy study of apparatus and methods used for testing the hardness of different materials with a view of determining their adaptability to the test of this property of slate has been made by Mr. R. Notvest, Consulting Engineer of the Structural Slate Co., and Mr. H. S. Booth of Western Reserve University. In this study they have arrived at the conclusion that the Hirschwald sclerometer, by certain modifications in its construction and a more definite means of measuring the hardness values obtained, affords a satisfactory means of determining the hardness of slate. They have pointed out certain features of the apparatus and method which appear to need further experimental research in order to arrive at a standard procedure. Further attention is being given to the subject of hardness, and in due time it is expected that a suitable method of test can be recommended.

Another property of slate which is of importance in some of its uses is that of toughness and it seems desirable to have a standard procedure for its determination. It is the intention of the committee to give this matter consideration in its future work.

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[1] See p. 756.—ED.

The weathering qualities of slate have, in general, been so satisfactory that little attention has ever been given to testing for durability. For materials from quarries which have been in operation over a long period this question would probably never be raised, but in exploiting new deposits from widely separated regions it appears desirable from the point of view of both producer and consumer to gain some information of the weathering qualities. Data on this subject are being collected and some experimenting is being done. It may be possible to report some progress along this line during the coming year.

With the growing interest in the work of the committee exhibited at the last meeting, the prospects of future accomplishment seem very good. It is especially gratifying to be able to report that hearty cooperation has been received from the producers of slate, which is of first importance to the success of the work.

The following is the result of the letter ballot of the committee on the submission of the tentative standards mentioned in this report:

Items	Affirmative	Negative	Not Voting
I. Proposed New Tentative Standards			
Tentative Method of Test for Water Absorption of Slate	13	0	1
Tentative Methods of Flexure Testing of Slate	13	0	1

This report has been submitted to letter ballot of the committee, which consists of 14 members, of whom 13 have voted affirmatively, none negatively, and 1 has refrained from voting.

Respectfully submitted on behalf of the committee,

D. W. Kessler,
Chairman.

D. K. Boyd,
Secretary.

Editorial Note

The proposed new Method of Test for Water Absorption of Slate; and the new Methods of Flexure Testing of Slate, were accepted for publication as tentative and appear on pages 754 and 756, respectively.

REPORT OF COMMITTEE E-1
ON
METHODS OF TESTING

Committee E-1 on Methods of Testing has held one meeting during the year, on February 20, 1925. The Advisory Committee held a meeting prior to the meeting of the main committee. In addition various sub-committees of Committee E-1 have held a number of meetings.

From much of the work recorded in this report it will be apparent that Committee E-1 on Methods of Testing is extending its endeavors in a study of methods from a broad viewpoint of a test as a type of test that may be made upon a wide variety of materials and in a study of the fundamental considerations that govern the various kinds of testing. This is apparent from the nature of the sub-committee reports presented and of the tentative standards that have been prepared and are being submitted either for acceptance by the Society for publication as tentative or as information. The standards submitted as information are the methods of flexure testing of metallic materials and the methods of torsion testing of metallic materials. The Tentative Specifications for Sieves for Testing Purposes, appended hereto, are submitted for acceptance as a tentative standard.

The major effort of the committee has been in the consideration of problems assigned to its sub-committees. These sub-committees are as follows:

Sub-Committee on Mechanical Testing;
Sub-Committee on Impact Testing;
Sub-Committee on Volatility;
Sub-Committee on Plasticity, Consistency, etc.;
Sub-Committee on Determination of Water;
Sub-Committee on Classification of Material According to Size.

The sub-committee organization is a close parallel to the classification of A.S.T.M. methods according to type of test which the committee made some few years ago to facilitate its work of reviewing the various methods of test. This classification was published in the report of Committee E-1 for 1924.[1] The several groups there shown have been assigned to the appropriate sub-committees. For example, the tests classified as mechanical tests made by application of stress-producing forces have been assigned to the Sub-Committee on Mechanical Testing, under the chairmanship of H. F. Moore. Those tests classified as mechanical tests involving such properties as consistency, plasticity, etc., have been assigned to the Sub-Committee on Consistency, under the chairmanship of W. E. Emley. Those tests covering the classification of material, including separation of materials, impurities, etc., have been assigned to the Sub-Committee on Classification of Material, recently broadened to a Sub-Committee on Size and Shape, under the chairmanship of F. G. Breyer. The tests for determination of water are under the jurisdiction of the Sub-Committee on Determination of Water (formerly Sub-Committee on Determination of Water in Bituminous Materials), under the chairmanship of W. H. Fulweiler. The tests for volatility are being studied by the Sub-Committee on Volatility, of which A. W. Dow is chairman.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 698 (1924).

There are a number of tests of a more or less special nature that cannot be readily assigned to the sub-committees mentioned. The review of many of these methods will consist in seeing that the requirements of the various methods are clearly and accurately stated; that no provisions are inconsistent with other methods of the Society, and that the form and arrangement of the methods are in accordance with the Society's requirements as given in the Regulations Governing the Form but not the Substance of Standards. This review, as mentioned in the report of the committee for 1924, has been placed in the hands of a special Editorial Committee consisting of C. L. Warwick, chairman, F. G. Breyer and W. H. Fulweiler, which committee reports to the Advisory Committee.

The review which the committee has in mind will of necessity take some time to complete. Committee E-1 re-affirms its position that it will not withhold indefinitely approval of such methods of test that standing committees might desire to see advanced to standard pending a completion of the review of these methods both as to form and the fundamental principles involved, but it will, however, reserve the right of reviewing the methods thus advanced to standard and submitting criticisms at some future time. Considerable progress was made during the year in the review of the miscellaneous methods by the Editorial Committee. A complete card index of the various methods of test is on file in the Secretary's office and has been kept up to date. This index was very helpful to the Editorial Committee as well as to the regularly organized sub-committees of Committee E-1.

With this at hand the Editorial Committee was able to reach some decisions as to the best means of taking up the several methods.

The committee found that there were many methods of test, the nature, apparatus and procedure of which were closely related and in some cases almost identical. It was felt that it was highly advisable to make a very careful study of these methods with a view to determine whether it would not be practical in some cases to write a single general method that would cover a number of the present methods which differ only in slight degree. The work of reviewing was started with the following types of determination:

Methods of test for solubility of organic solvents and for refractive index.

Methods of test for iodine number, polymerization, saponification, unsaponifiable matter, determination of acidity.

Methods of freezing and thawing and colorimetric tests.

These methods are being studied by the individual members of the Editorial Committee. It seemed desirable to call in men outside of its membership for certain methods and F. H. Jackson was asked to assist the committee in the study of the present methods for specific gravity, and A. C. Fieldner was asked to assist in the study of methods for fusibility of coal ash and refractories. Mr. Jackson has made considerable progress in his review of the methods of test for specific gravity and Mr. Fieldner has submitted an analysis of the methods of test for fusibility of coal ash in comparison with the methods of test for melting point of refractory brick. This analysis is now being given consideration by Committee C-8 on Refractories. In the light of this analysis perhaps some unification can be accomplished in these two tests.

The review of the various methods carried out by the Editorial Committee resulted in the committee presenting a number of recommendations to Committee E-1 as follows:

1. That references in standards should be to A.S.T.M. methods only. If the A.S.T.M. has prepared no corresponding method then the method should be written up as an A.S.T.M. standard so as to appear in the publications of the Society, giving due credit to the source from which the material is taken.

Many methods at present refer to publications of other societies or to textbooks, hand books, etc. This makes it difficult for the methods to be followed since the other publications referred to may not be available. Such references should accordingly be eliminated and reference made only to the standards of the Society. Such standards would be immediately accessible and the method in question accordingly would be complete. It is to be understood that where a reference is merely incidental, as for example, a reference to another publication for directions in more complete detail, such references might be retained.

2. Methods should have a more complete section on scope. This should be a descriptive paragraph stating exactly what property of a material the method is intended to determine and what significance this property may have. It should set forth the limitations of the method and why the results obtained may sometimes be misleading. Such a paragraph would be most illuminating and would enable an investigator to use the method more intelligently.

3. The use of adequate diagrams or drawings will often aid materially in clarifying descriptions of complicated apparatus and may frequently permit more concise descriptions in the text. Photographs of apparatus are desirable if they can be included without unduly emphasizing the apparatus of any particular manufacturer.

4. Standards should be more specific as to the method or methods that are to be employed.

Certain standards enumerate several instruments or methods that may be employed in making a determination, perhaps concluding the series with "or any other accurate method," leaving it to the judgment of the operator as to whether some other method is equally accurate. This, of course, leaves the requirement quite open and nothing has been accomplished in the way of writing a definite procedure that would be binding.

5. Instead of numerous descriptions for a certain determination appearing in various standards, have one clear-cut general method complete with definitions and descriptive paragraph, which method could be referred to in the several standards.

These recommendations were given consideration at a meeting of Committee E-1 and were approved for publication in the annual report of the committee. They are accordingly presented and will be referred to the standing committees of the Society. It is believed that if the suggestions are followed our methods will be more serviceable than as at present prescribed.

SUB-COMMITTEE ON MECHANICAL TESTING

The Sub-Committee on Mechanical Testing (H. F. Moore, chairman) until recently was entitled Sub-Committee on Mechanical Testing of Metallic Materials. Accordingly its activities were confined to a study of tests as related to metallic materials. The tentative methods prepared by this committee and submitted in 1924 were carefully reviewed, and a number of criticisms that had been received were considered. As a result of this further study, the sub-committee is recommending a number of revisions in the tentative standards. These revisions are given herewith and Committee E-1 recommends that these revisions be accepted and the tentative standards as revised continued as tentative.

Tentative Definitions of Terms Relating to Methods of Testing (E 6 - 24 T).[1]—The following revisions are recommended:

Under the definition for the term "stress" add the following sentence to the note:

> "It is customary to compute stress on the basis of the original dimensions of the cross-section of the body."

Under the definition for the term "tensile strength" add the following sentence to the note:

> "Tensile strength is computed from the maximum load carried during a tension test and the original cross-sectional area of the specimen."

In the definitions for the terms "elastic limit," "tensile strength" and "compressive strength" change the word "withstanding" to "developing." The idea of this change is that the stress is a force which is developed within a body rather than a force which is withstood.

The definition for "tensile strength" also appears in the Tentative Methods of Tension Testing of Metallic Materials, the definition for "elastic limit" in the Tentative Methods of Tension Testing and of Compression Testing of Metallic Materials, and the definition for "compressive strength" in the Tentative Methods of Compression Testing of Metallic Materials. The definitions appearing in these tentative standards should be revised to agree with the change given above.

Change the definition of "compressive strength" from its present form: namely,

> "The maximum stress which a material that fails in compression by a shattering fracture is capable of withstanding."

to read as follows:

> "Compressive strength is the maximum compressive stress which a material is capable of developing.
>
> "NOTE.—In the case of a material which fails in compression by a shattering fracture the compressive strength has a very definite value. In the case of materials which do not fail in compression by a shattering fracture the value obtained for compressive strength is an arbitrary value depending upon the degree of distortion which is regarded as indicating complete failure of the metal."

In addition to the revision of the definition of "elastic limit" given above, it is recommended that the first two paragraphs of the note reading as follows:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 1109 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 711.

"Since experience does not indicate any appreciable difference between the elastic limit and the limit of proportionality of stress (sometimes called the proportional limit), the determination of the stress at the limit of proportionality of stress to strain is regarded as an accurate determination of the elastic limit.

"It is obvious that the values obtained in tests for determining the elastic limit will depend on the delicacy of methods and instruments used. It becomes necessary, therefore, that in any test the method used in obtaining the elastic limit be clearly stated. The following methods are in common use for determining a value designated as the elastic limit:"

be replaced by the following:

"NOTE.—In certain specifications of the Society the term 'elastic limit' is used to designate a value obtained by a test method which does not involve the release of stress during a test, but which does involve the determination of a limiting load at which there occurs an appreciable change in rate of strain with respect to stress. This limit is frequently given the name 'proportional limit' (or 'proportional elastic limit'), and the following definition may be given:

"*Proportional Limit.*—The greatest stress which a material is capable of developing without a deviation from the law of proportionality of stress to strain (Hooke's Law).

"It is a matter of experience with many materials, especially with many metallic materials, that using ordinary methods of testing, the values found for elastic limit by means of observations of permanent deformation (set) after release of stress do not differ widely from the values found for proportional limit. Since the determination of proportional limit is much more readily made than is the determination of elastic limit it is customary to accept the proportional limit as equivalent to the elastic limit for such materials.

"Using any known method, the value determined either for porportional limit or for elastic limit is somewhat arbitrary; the value depends on various test conditions, among them precision of apparatus, the scales to which stress-strain diagrams may be plotted, and the tolerance allowed before it is judged that there exists an appreciable deviation from the law of proportionality of stress to strain or an appreciable permanent deformation (set) upon release of stress.

"In connection with this note on elastic limit and proportional limit, it seems proper to consider the action of a material under steadily increasing stress, and to discuss briefly five stages which may be distinguished between the first application of stress and fracture of the metal. Not all materials show all five stages, and for a material the end of one stage and the beginning of the next stage are not always marked.

"In the first stage (*OA*, Fig. 1), with the use of measuring instruments of ordinary sensitiveness, deviations from the law of proportionality of stress to strain (Hooke's Law) will not be detected, nor will there be detected set after release of stress.

"In the second stage (*AB*, Fig. 1) as stress increases there is detected an appreciable and growing deviation from the law of proportionality of stress to strain. This is usually accompanied by an appreciable and growing permanent deformation (set) after release of stress, although for some materials (*e.g.*, rubber) measurable permanent deformation may not occur after release of stress exceeding the proportional limit. Values for elastic limit and for proportional limit are found in this second stage.

"The third stage (*BC*, Fig. 1) is characteristic of ductile metals. In this stage, strain increases without any increase of stress, and in some cases increase of strain is accompanied by an actual decrease of stress. The yield point is found at the beginning of this stage (at *B*, Fig. 1).

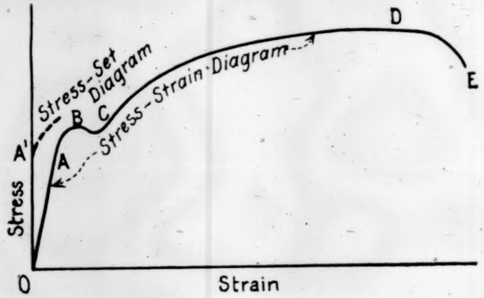

FIG. 1.

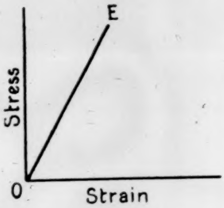

FIG. 2.

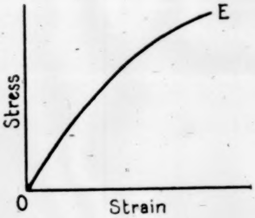

FIG. 3.

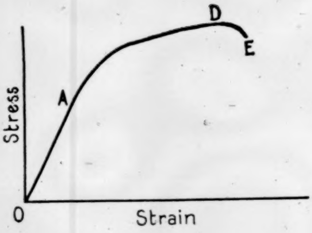

FIG. 4.

"In the fourth stage (*CD*, Fig. 1) stress and strain both increase up to the maximum stress carried (*D*, Fig. 1, which gives the tensile strength in a tension test of a material).

"In the fifth stage (*DE*, Fig. 1) strain increases while stress diminishes from the maximum value (*D*, Fig. 1) to fracture (*E*, Fig. 1). In considering each of these stages it is important to note that stress is computed on the basis of original cross-section of specimen.

"Many materials, especially brittle materials, do not exhibit all the above-mentioned stages. Some (*e.g.*, glass and fused quartz) show only the first stage. Fig. 2 gives a representative stress-strain diagram for such materials. Some materials (*e.g.*, cast iron) show only the second stage, and Fig. 3 shows a typical stress-strain diagram for such materials. Some materials (*e.g.*, medium-hard steel) do not show the third stage, and the second stage merges into the fourth stage. Fig. 4 shows a typical stress-strain diagram for such materials. The fifth stage is absent in many materials; it is, in fact, characteristic of ductile metals only.

"The following methods of test are, strictly speaking, methods of determining proportional limit with various degrees of precision. However, the values determined by these methods may, for certain materials, be regarded as satisfactory and reliable values for the elastic limit, and they are therefore so used in certain specifications of the Society."

Tentative Methods of Tension Testing of Metallic Materials (*E 8 - 24 T*).[1]—The following revisions are recommended:

Section 4.—Change the footnote to this section to read:

"Attention is called to Section 7."

Section 13.—At the end of the next to the last sentence, after the words "exceed 0.01 in." add "for any position of the movable head within the range of movement during test." In the next sentence, change the words "movable head" to the single word "heads."

Section 14.—Change the second paragraph to read as follows by the addition of the italicized words and the omission of those in brackets:

"[The cross-head speed of the testing machine shall be such that the beam of the testing machine can be kept balanced,] *The speed of head of the testing machine shall be such that the load can be accurately weighed*.........."

Change the footnote to read as follows:

"The speeds given here are probably too high for satisfactory use in testing non-ferrous metals."

Section 18.—Change the first two sentences under Method II to read as follows by the addition of the italicized words and the omission of those in brackets:

"In Method II [for determining the yield point, one observer keeps the beam of the testing machine balanced, and another] *an* observer with a pair of dividers watches for visible elongation between two [gage] *section* marks on the

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 1081 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 683.

[specimen] ***specimens.*** When visible stretch is observed [the fact is at once reported to the man operating the beam of the testing machine,] the load at that instant is noted, and the stress corresponding to the load is taken as the yield point."

Section 20.—Add to the note under this section the following sentence:

"This method is not applicable to brass specimens."

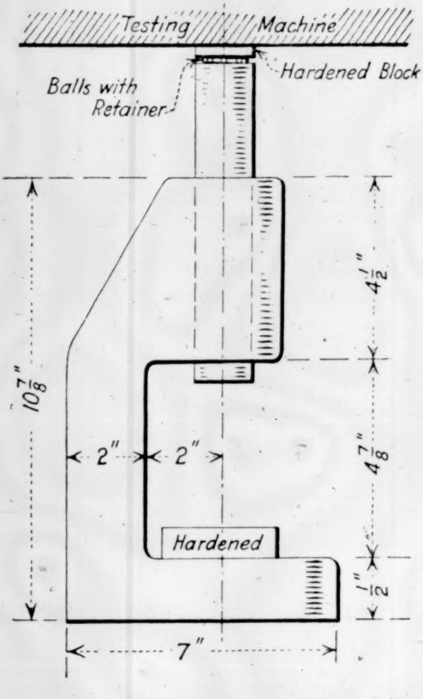

FIG. 5.—Compression Tool.

Tentative Methods of Compression Testing of Metallic Materials (E 9 - 24 T).[1]—The following revisions are recommended:

Section 3.—Change the fourth sentence to read as follows by the addition of the italicized words and the omission of the word in brackets:

"Long specimens are [used] ***best adapted*** for determining the modulus of elasticity in compression of metallic materials."

Omit the present Footnote 2 to this section reading as follows:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 1094 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 696.

"It has been suggested in discussion that the medium-length specimen may be used for determining modulus of elasticity. See discussion of the Report of Committee E-1."

Section 4.—Change the third paragraph from its present form: namely,

"Medium-length specimens: $d \pm 1$ in. ± 0.01 in., $h = 3$ in.[1]"

to read as follows:

"Medium-length specimens: $d = 0.798$ in. ± 0.01 in., $h = 2\frac{3}{8}$ in. or
$d = 1$ in. ± 0.01 in., $h = 3$ in. or
$d = 1\frac{1}{8}$ in. ± 0.01 in., $h = 3\frac{3}{8}$ in."

Omit the footnote to this section reading as follows:

"It was suggested in discussion that for medium-length and long specimens d might be $1\frac{1}{8}$ in. Another suggestion was that d be made 0.798 in. See discussion of the Report of Committee E-1."

Section 8.—Change Footnote 1 to this section to read as follows:

"The device shown in Fig. 5 has been successfully used in compression tests of metallic specimens whose ends were machined with extreme care. It will be noted that this device does not involve a spherical seated compression block bearing directly on the test specimen. This device or one similar has been successfully used in the British National Physical Laboratory and in the laboratories of the U. S. Air Service at McCook Field, Dayton, Ohio."

Section 15.—Change the definition of "compressive strength" from its present form: namely,

"The maximum stress which a material that fails in compression by a shattering fracture is capable of withstanding."

to read as follows:

"Compressive strength is the maximum compressive stress which a material is capable of developing.

"NOTE.—In the case of a material which fails in compression by a shattering fracture the compressive strength has a very definite value. In the case of materials which do not fail in compression by a shattering fracture the value obtained for compressive strength is an arbitrary value depending upon the degree of distortion which is regarded as indicating complete failure of the metal."

Omit the second paragraph of this section.

Tentative Methods of Brinell Hardness Testing of Metallic Materials (E 10 - 24 T).[1]—The following revisions are recommended:

Table I.—Insert a paragraph to read as follows:

"The values given in this table for hardness numbers are merely solutions of the equation given on the preceding page. They do not imply that Brinell tests are feasible on materials of a hardness indicated by the highest values in the table."

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 1099 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 701.

Section 1.—Change from its present form: namely,

"The micrometer microscope or other measuring device used for the measurement of the diameter of impression shall be capable of measuring the diameter of the impression within 0.01 mm. (0.0004 in.).

to read as follows:

"The divisions on the micrometer scale of the microscope or other measuring device used for the measurement of the diameter of impression shall be such as to permit the direct measuring of the diameter to 0.1 mm. and the estimation of the diameter to 0.01 mm."

Section 2.—Change Paragraph (*a*) to read as follows by the addition of the italicized words:

"The balls used in Brinell hardness testing shall be of hardened steel *or other equally hard material.*"

Change the footnote to Paragraphs (*b*) and (*c*) to read as follows:

"In testing softer metals, it is not, of course, necessary to have balls of this extreme hardness. However, a ball to be suitable for use should withstand pressure against the hardest material to be tested without showing a permanent change in diameter greater than 0.0025 mm."

Section 3.—Change the first paragraph to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"The standard ball for Brinell hardness testing shall be 10 mm. (0.3937 in.) in diameter with a deviation from this value of not more than [0.0025 mm. (0.0001 in.)] *0.01 mm.*(*0.0004 in.*) in any diameter, *but the diameter of any ball measured at various points shall be constant within a tolerance of ±0.0025 mm. (0.0001 in.).*"

Section 4.—Add a sentence to read as follows:

"In any event the thickness of the specimen shall be at least 10 times the depth of impression."

Omit the footnote reference to this section.

Section 9.—In the note under "Brinell Hardness Tests in which Depth of Impression is Measured in Place of Diameter," change the second sentence to read as follows by the addition of the italicized words:

"The depth of impression is usually determined from the *relative* motion of the plunger carrying the ball *and the specimen.*"

Section 11.—In the first sentence, after the words "proving levers" add the words "or by an elastic calibration bar or spring."

Section 15.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> "A Brinell hardness testing machine is acceptable for [general] use [if] *over a loading range within which* its load-measuring device is correct within 3 per cent."

In addition to the revisions given above, the sub-committee submits the following new method as a more precise method for determining elastic limit. This is presented by Committee E-1, not as a revision of the present Tentative Definitions of Terms Relating to Methods of Testing (E 6 – 24 T) and of the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 24 T), to which the method would be added if finally accepted, but rather as information to elicit comment and criticism:

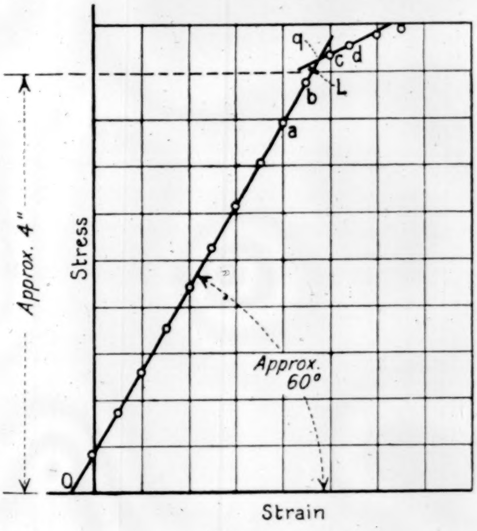

Fig. 6.

PROPOSED ADDITIONAL METHOD FOR DETERMINING ELASTIC LIMIT

Method III is a more delicate method than Method II. In Method III the elastic limit shall be taken at that stress for which there can be detected a departure of the stress-strain diagram from a straight line. The stress-strain diagram may be either an autographic or semi-autographic diagram drawn during the progress of the test or a diagram plotted from observed data.

In either case a strainometer shall be used which can indicate a strain as small as 0.0001 of the gage length for tension or compression, and a correspondingly small strain for flexure or torsion; the testing machine used shall be capable of indicating a change of load as small as 0.004 of the estimated load at the elastic limit. During the test, the load shall be applied so as to give ten approximately equal increments of strain (as indicated in Fig. 11 (*b*) of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 - 24 T), up to the estimated value of the elastic limit and the test shall be continued with at least three increments of strain beyond the elastic limit.

The stress-strain diagram should be plotted to such a scale that the ordinate of stress at the elastic limit shall be approximately 4 in. and the initial (straight) portion of the diagram shall make an angle of approximately 60 deg. with the horizontal.

If the location of the point of departure from a straight line is at all uncertain, as is the case shown in Fig. 6, in which the outline of the diagram between *b* and *c* is uncertain, the following method of locating that point of departure is recommended. Through the two points *c* and *d*, (Fig. 6) which are the lowest two stress readings which are distinctly off the straight-line portion of the diagram, draw a straight line intersecting *Oa* at *q*. Locate the elastic limit at *L* midway between *q* and *b*, the observed point of highest stress on the straight-line portion of the diagram.

The sub-committee is presenting two new methods of test entitled Methods of Flexure Testing of Metallic Materials and Methods of Torsion Testing of Metallic Materials. These methods have been before the sub-committee for a year or more. They have been approved by Committee E-1 and are being published as appended hereto as information. The Methods of Flexure Testing cover the tests usually made on cast iron but are applicable to other metallic materials as well. The Methods of Torsion Testing are intended primarily for determining the shearing properties of a metal.

The sub-committee is now turning its attentions to the testing of thin sheet material. The importance of satisfactory tests for this material was emphasized at the last annual meeting and a special sub-committee has accordingly been appointed to take this important study in hand to consist of the following: H. C. Knerr, H. A. Anderson, R. L. Templin and C. H. Marshall.

The broadening in scope of the sub-committee as indicated by the change in title will be accompanied by a corresponding increase in personnel so as to make the sub-committee sufficiently representative to consider the methods of mechanical testing as applied to all engineering materials. This broadening of the interests of the sub-committee may ultimately lead to the broadening of the present methods as prepared by this sub-committee so as to apply to materials other than metallic materials. The considerations involved in the mechanical testing of all materials have much in common and this may be reflected in the nature of the standards formulated by this sub-committee in the future.

SUB-COMMITTEE ON DETERMINATION OF WATER

The Sub-Committee on Determination of Water, under the chairmanship of W. H. Fulweiler, has made a careful study of the various methods for determining water and an analysis has been prepared and is given in the report of the sub-committee that is appended hereto. The scope of the sub-committee has been enlarged as is indicated by the change in title from Sub-Committee on Determination of Water in Bituminous Materials to Sub-Committee on Determination of Water. A corresponding increase in personnel will be made.

SUB-COMMITTEE ON CLASSIFICATION OF MATERIAL ACCORDING TO SIZE

In the report of Committee E-1 for 1924 a proposed series of testing sieves was presented as information. The Sub-Committee on Classification of Material According to Size has continued its efforts to establish a standard screen scale. This sub-committee is under the chairmanship of F. G. Breyer, but J. P. Hubbell has acted for Mr. Breyer in the capacity of chairman. The sub-committee has made considerable progress during the past year and Committee E-1 owes its appreciation to Mr. Hubbell for his efforts in advancing the work. The sub-committee for the present has confined its attentions to a study of sieve specifications and methods of classifying materials by means of sieves. The proposed series published last year was brought to the attention of a number of interested parties and as much publicity as possible has been given to the activities of the sub-committee in order to secure the advice and cooperation of other organizations in the interest of having one standard screen scale adopted if possible. Invitations were extended to outside organizations to attend the meetings of the sub-committee. These included Ludlow-Saylor Co., W. S. Tyler Co., Multi-Metal Co., Newark Wire Cloth Co., Arthur H. Thomas Co., American Water Works Association, Manufacturing Chemists Association, Scientific Materials Co., Bureau of Mines, Eimer & Amend, Institute of Paint and Varnish Research, American Institute of Mining and Metallurgical Engineers, and the Rubber Association of America. The sub-committee has been in constant touch with the manufacturers of testing sieves and at a meeting held in January representatives of several of the manufacturers were present. At this meeting the proposed series of requirements was formally approved and actions were taken leading to the preparation of complete specifications for sieves for testing purposes, including not only requirements for the cloth but the requirements for the frames, the methods of testing the cloth to determine whether it conforms to the specifications, and the methods of labeling the sieves. These specifications are appended hereto.[1]

One important innovation was introduced that is worthy of comment. This is the method used to designate the sieves. In the series recommended in the proposed specifications, the sieves are designated by the size of the opening in microns. In the past, two common methods have been used to designate testing sieves, first, mesh, and, second, arbitrary numbers corresponding closely to the mesh. The most practical method of determining the size of opening in a fine sieve is to measure the diameter of the wire and count the number of wires per inch or other lineal unit. From this method of measurement the mesh designation arose. In its use, however, the fact that the size of the wire, as well as the number of wires per inch, controlled the size of opening, was generally forgotten, so that sieves of widely different openings but of the same number of wires per inch were given the same designation. The arbitrary number system was devised to take care of this, the No. 200 sieve, for instance, being a 200-mesh sieve of some specific wire size and no other. This method of designation, however, was subject to the drawback of all methods depending upon arbitrary figures. It could only work satisfactorily if everyone accepted it. There is a further drawback to both of these methods in that, unless one is thoroughly familiar with the appearance of each sieve, the designation gives no direct idea of the size of material which would pass or be retained. The new micron designation seems to answer all of these objections. It labels the sieve according to that part of it which is of real importance, namely, the opening, and it gives at the same time a direct measure of the size of the material which passes through or is retained on the particular sieve in question.

The reasons for fixing upon the micron rather than some other unit of measurement were several. To mark the finer sieves in terms of either inch or millimeter is difficult in that it would involve the use of decimals to a considerable number of places, but the micron on the other hand permits the use of whole numbers for all sizes, and furthermore this method of designation can be easily extended to sizes less than sieve size. It has the further advantage of getting away completely from the methods of designation already in use and at the same time giving an exact idea of the size of opening directly. It is a scientific designation for a scientific instrument and should help to bring about the conception of a sieve as an accurate measuring instrument. While some education may be necessary to bring about the general use of this designation, a large number of those using sieves, such as chemists, are already familiar with the use of the micron unit. For the benefit of those unfamiliar with the metric unit the inch equivalent may still be given on the sieve label, and provision for this is made in the recommendations of the proposed specifications.

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[1] See p. 850.—ED.

If the size of opening of a sieve is kept in mind, the present unsatisfactory condition in industries where reports of mechanical separation by means of sieves are involved, would be considerably clarified. So long as the properties of the sieves used are known, then results reported by mechanical analysis using those sieves may be intelligently interpreted regardless of the actual sieves employed. It is merely a question of properly plotting the test data. The sub-committee accordingly recommends that the standing committees give more prominence to the actual size involved when giving the size of particle. For example, material could be referred to as passing a sieve having a certain size of opening and retained upon a sieve having some other designated opening. A typical requirement might read: "Material not less than 80 per cent of which passes a 300-micron sieve." The proposed designation goes far towards accomplishing the desired result, and the universal adoption of the proposed specifications would be the ideal so far as testing by means of sieves is concerned. However, even without such universal adoption, the result would in great measure be accomplished through the recognition that the size of opening is the important consideration.

Committee E-1 approves the recommendation of the sub-committee as to the emphasis that is to be placed on the size of opening in the standards of the Society and wishes to bring this recommendation to the attention of the standing committees interested. It further approves the acceptance by the Society of the proposed Tentative Specifications for Sieves for Testing Purposes appended hereto.

It is hoped that these specifications will receive general acceptance. The preliminary drafts were brought to the attention of other interested organizations and a number of favorable comments have been received. The committee will continue its policy of cooperation with these organizations.

As mentioned elsewhere in this report, the title of the sub-committee has been changed to Committee on Size and Shape. As this would indicate, the committee expects to take under consideration other means of classification besides those involving sieves, its next field possibly being that of sub-sieve sizes.

SUB-COMMITTEE ON VOLATILITY

The principal work of the Sub-Committee on Volatility, of which A. W. Dow is chairman, has been to try to harmonize the various methods of distillation. The work of the sub-committee has resulted in a careful re-examination being made by the standing committees concerned of the distillation methods under their jurisdiction. This may result in some changes in the requirements for the apparatus employed.

A review is being made of the numerous loss on heating tests and tests for volatile matter that come under the province of the sub-committee. It is expected that a report on this subject will be forthcoming during the year.

SUB-COMMITTEE ON CONSISTENCY

The Sub-Committee on Consistency, under the chairmanship of W. E. Emley, has been endeavoring to prepare a definition of the term "consistency." While no final action has as yet been taken, a definition for consistency has been prepared and is now being circularized to elicit discussion.

The committee still has before it the review of the various methods of the Society dealing with the determination of consistency and of allied properties.

SUB-COMMITTEE ON IMPACT TESTING

The Sub-Committee on Impact Testing, under the chairmanship of E. B. Smith, has given consideration during the year to the possibilities of preparing a recommended practice for the impact testing of metals. The impact test, although not yet standardized, is being used for a variety of purposes and is applied to a number of materials. It might be well to have a recognized recommended practice which could be used as a relative test and further research might result in the preparation of a fundamental test.

As a preliminary step the sub-committee expects to make an analysis of the papers presented as a part of the Symposium on Impact Testing held in 1922.[1] There were considerable data of value on record in the papers presented at that time. Much could be gained by making this material more readily accessible. It was hoped that an analysis giving in summarized form the general conclusions that could be drawn from the statements there made could be included with this report but the analysis is not yet available.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 5 (1922).

The analysis of the Symposium may make evident along what lines the information is lacking and the most fruitful means of research that should be carried out. A special committee has been appointed to lay out a program for comprehensive research, which plan should be laid before the Society's Committee on Correlation of Research. This special committee has been appointed to consist of the following: E. H. Dix, Jr., W. H. Fulweiler, F. C. Langenberg, T. D. Lynch, E. B. Smith and H. L. Whittemore. It is planned to hold a meeting of this special committee in conjunction with the annual meeting of the Society.

The recommendations that appear in this report have been submitted to letter ballot with the following results:

Items	Affirm-ative	Neg-ative	Not Voting
I. REVISIONS OF TENTATIVE STANDARDS			
1. Tentative Methods of Tension Testing of Metallic Materials (E 8 - 24 T)	26	0	19
2. Tentative Methods of Compression Testing of Metallic Materials (E 9 - 24 T)	26	0	19
3. Tentative Methods of Brinell Hardness Testing of Metallic Materials (E 10 - 24 T)	20	0	25
4. Tentative Definitions of Terms Relating to Methods of Testing (E 6 - 24 T)	30	0	15
II. PROPOSED NEW TENTATIVE STANDARD			
5. Tentative Specifications for Sieves for Testing Purposes	29	0	16
III. SUBMITTED AS INFORMATION			
6. Proposed Methods of Flexure Testing of Metallic Materials	27	0	18
7. Proposed Methods of Torsion Testing of Metallic Materials	26	1	18
8. Additional Method for Determination of Elastic Limit	25	2	18

This report has been submitted to letter ballot of the committee with the following result: Advisory Committee, 8 affirmative, 0 negative, 0 not voting; total, 8; Standing Committees, 24 affirmative, 0 negative, 14 not voting; total, 27.

Respectfully submitted on behalf of the committee,

J. A. CAPP,
Chairman.

C. L. WARWICK,
Secretary.

EDITORIAL NOTE

The proposed revisions of the Tentative Methods of Tension Testing of Metallic Materials; of Compression Testing of Metallic Materials; of Brinell Hardness Testing of Metallic Materials; and of the Tentative Definitions of Terms Relating to Methods of Testing, were accepted. The tentative methods and definitions of terms as thus revised appear on pages 854, 867, 872 and 879, respectively.

The proposed new Specifications for Sieves for Testing Purposes were accepted for publication as tentative and appear on page 850.

REPORT OF SUB-COMMITTEE ON DETERMINATION OF WATER

There is probably no constituent whose determination is shrouded in more doubt or uncertainty than the apparently very simple determination of the amount of water or moisture in our ordinary materials.

The most general method for the determination of water consists in heating a weighed sample of material in a drying oven at some elevated temperature, usually 100 to 105° C. for several hours and calling the loss of weight thus incurred, moisture or water. The results obtained in this manner may be either much too high or much too low, depending upon the material tested and other conditions. Such a method of determination, *i.e.*, by loss of weight, is known as an indirect method.

In bituminous materials such as petroleum tars, etc., water is usually determined directly by distilling the material, collecting the water present and determining the quantity from the mass or volume. With certain materials, this distillation is difficult or may even be impossible due to foaming, decomposition, etc.

The two principal difficulties that arise in methods for the determination of water are due to (1) the diversity of forms in which water may occur and (2) the stability of the material under test.

I. Diversity of Occurrence.—

In any given material, the water present may occur in one or more of the following forms:

1. As free surface water.

If the physical conditions were suitable such water might be wiped free from the surface of the material. Under other conditions of subdivision, where the surface is very greatly extended and in the form of fine interstices, the water may be so tightly held as the result of physical forces that it cannot be readily driven off, even if exposed to relatively high temperatures.

2. As water of hydration.

Depending upon the materials, some of this water may be readily given off at ordinary or slightly elevated temperatures, but other portions require ignition up to a red heat before all of this water is driven off.

3. As water of crystallization.

Again, some proportion of such water is readily given off even at the ordinary temperatures, while other portions may require ignition or in fact may even resist strong heating.

4. In solution.

With certain materials, water may exist as a true solution and frequently will require chemical means for its separation. In certain organic liquids in which water is relatively insoluble, the small amount present may be readily removed by the passage of a dry gas but this method will not suffice where there appears to be any considerable amount of solubility.

5. As a dispersed or dispersing phase in colloidal solution or suspensions.

This class of materials usually gives a great deal of trouble with the accurate determination of moisture. It is usually necessary to use some means to "break" the emulsion which will result in setting free the greater portion of the water but small portions are usually very tightly held by surface forces.

II. Stability of Material Under Test.—

The second difficulty in the determination of water is that which is due to the stability or instability of the material under test when exposed to elevated temperatures. This instability may result in decomposition, the result of which is the production of water by chemical reaction, from existing constituents by oxidation whereby water may be produced by combination with the oxygen from the surrounding atmosphere; by oxidation in which oxygen is taken up from the surrounding atmosphere, thus changing the weight of the water-free material; and by volatilization.

Under this heading, we might also consider the mobility of the material even at elevated temperatures, whereby it is difficult for the free water present to escape from the material. This difficulty is usually surmounted by reducing the thickness of the material exposed by greatly extending the surface by mixing with kieselguhr, sand, beads or absorbing in filter paper.

In addition to the technical difficulties, which in many cases can be surmounted in a research laboratory at the considerable expenditure of time and by the use of special devices and apparatus, we have the fundamental requirement for a technical determination that it should be secured reasonably quickly and in a manner that will permit duplication of the results by different laboratories. While it is always desirable that the method used should be fundamentally correct, in the case of many technical analyses, the time required to secure such theoretically correct results and the complication due to the method and apparatus required are such that the slight gain in accuracy that is secured does not appear to compensate for the increased difficulty in carrying out the determination. In other words, in many technical determinations we use empirical methods which the industries find to be satisfactory.

A review of the standards and tentative standards of the Society shows that they, with the exception of the method described in the Standard Specifications for Turpentine (D 13 - 24), where the free water in the sample is merely decanted, the methods may be divided into two general classes, direct methods where the water existing in the sample is actually separated out and determined as such, and indirect methods where the water, driven off from the sample is obtained by difference. The methods are listed in Tables I and II.

TABLE I.—MOISTURE DETERMINED BY INDIRECT METHODS.

Serial Designation	Material	Apparatus	Temperature	Time	Atmosphere	Ventilation	Remarks
D 29	Shellac..........	Oven	38 - 43° C.	3–6 hours	Air	Well ventilated	
C 26	Gypsum.........	"	45° C.	2 "	"	None	Free water
D 198 T	Timber.........	"	98±3° C.	To constant weight	"	"	
D 143 T	Timber.........	"	100° C.	To constant weight	"	"	
D 202 T	Insulating paper.	"	100 - 105° C.	To 0.2 per cent	"	"	
D 39	Fabric..........	"	105 - 110° C.	To constant weight	"	"	
D 49	Red Lead.......	"	105 - 110° C.	2 hours	"	"	
D 126	Chrome pigments	"	105 - 110° C.	2 hours	"	"	
D 186 T	Titanium pigm'ts	"	105 - 110° C.	2 hours	"	"	
D 22	Coal...........	"	Atmos.+(10 - 15° C.)	To 0.1 per cent	"	Yes	Air drying
D 22	Coal...........	"	104 - 110° C.	1 hour	Dry air	"	Final moisture
D 37	Coke...........	"	104 - 200° C.	To constant weight	Air	None	Total moisture
D 37	Coke...........	"	104 - 110° C.	1 hour	"	"	Moisture

TABLE II.—MOISTURE DETERMINED BY DIRECT METHODS.

Serial Designation	Material	Method
D 96	Petroleum products....................	Solution in benzol and centrifuge
D 38	Creosote....................	Direct distillation from copper still
D 43 T	Creosote oil primer....................	" " " " "
D 95 - 24	Petroleum products....................	Distillation with solvent. Method D 95 - 24
D 42 T	Pitch for waterproofing....................	" " " " "
D 104 T	Road tar....................	" " " " "
D 105 T	Road tar....................	" " " " "
D 106 T	Tar cement....................	" " " " "
D 107 T	Tar cement....................	" " " " "
D 108 T	Tar for surface treatment....................	" " " " "
D 109 T	Tar for surface treatment....................	" " " " "
D 110 T	Tar cement....................	" " " " "
D 111 T	Tar cement....................	" " " " "
D 112 T	Pitch fillers....................	" " "
D 128 T	Grease....................	" " "
D 145 T	Pitch for waterproofing....................	" " "
D 146 T	Fabric for saturation....................	" " "
D 158 T	Gas oils....................	" " "
D 200 T	Pitch....................	" " "
D 201 T	Pitch....................	" " "

It will be noted that those methods in which the material is heated to a given temperature for a definite length of time or to what is known as constant weight, are all applied to solid materials.

In the second group of methods, known as direct methods, the water is separated from the material and determined as such either by dilution with a solvent and centrifuging as in Method D 96; by simple distillation as in the case of Methods D 38 for creosote oil; or

TABLE III.—SUMMARY OF VARIOUS METHODS FOR DETERMINATION OF MOISTURE.

DIRECT METHODS

1. Separation by gravity.
2. Separation by centrifugal force.
3. Dilution with solvent and centrifuge.
4. Drying in current of gas at ordinary temperature with recovery of water.
5. Drying in current of gas at elevated temperature with recovery of water.
6. Heating with recovery of water.
7. Distillation at high temperatures.
8. Distillation with a solvent immiscible with water.

INDIRECT METHODS

A.—CHEMICAL METHODS

9. Formation and measurement of hydrogen from sodium.
10. Formation and measurement of acetylene from calcium carbide.
11. Formation and measurement of methane from Grignard's Reagent.
12. Change in strength of standard acid.

B.—PHYSICAL METHODS

At Elevated Temperatures.—

13. By direct heating at moderate temperatures.
14. By ignition at very high temperatures.
15. Heating in high boiling fluids.
16. Heating with a volatile fluid.
17. Oven drying in normal atmosphere.
18. Oven drying in a ventilated oven.
19. Oven drying in oven with regulated atmosphere.
20. Vacuum oven.
21. Vacuum desiccator.

At Normal Temperatures.—

22. Vacuum desiccator.
23. Extraction with a solvent and determination of change in solvent.
24. Fall in temperature on passage of current of air.
25. Rise in temperature on mixing with anhydrous $MgSO_4$.
26. Rise of temperature (in acids) on addition of water.
27. Color variation either with or without use of dyes.
28. Electrical conductivity of solids as paper and pulp, soil.
29. Electrical conductivity of solution and emulsions.
30. Change in boiling point of material.
31. Change in melting point of material.
32. Change in refractive index.
33. Change in specific gravity.
34. Critical temperature of solution.
35. Change in volume on mixing with an absorbent.

by distilling the material with an immiscible solvent and measuring the water which is carried over as in Method D 95 – 24.

Although there are three or four different types of determinations used in our present standards, we cannot but feel that we are making considerable progress in standardization if we will but consider the large number of methods for determination of water that have been proposed and in many cases are actually in use for this determination. Table III gives a very brief summary of a few of these.

It will be noted that many ingenious ideas have been utilized in order to avoid the difficulties which continaully arise in the determination of water with certain materials.

A study of the literature indicates that in many cases where water has been determined by heating the material at definite temperatures, say of the order of 100 to 105° C., this method does not determine the actual water content but in many cases the results thus obtained due to the action of conflicting forces may represent to a fairly high degree of accuracy this figure.

The determination of the moisture in coal by heating has received a very great deal of study and illustrates the point that while the standard method is known to be subject to certain fundamental errors, yet the results obtained appear to be very close to the true amount of water present as determined by the more elaborate direct methods. This matter was gone into at considerable length by a sub-committee appointed by the Eighth International Congress of Applied Chemistry to which reference should be made for much interesting data.

During the past two years, the sub-committee assisted in the development of the Method D 95 – 24, which was adopted jointly by Committees D-2 on Petroleum Products and Lubricants, and D-4 on Road and Paving Materials and accepted by Committees D-7 on Timber and D-8 on Waterproofing Materials, for the determination of water in a number of bituminous materials. As shown in Table II, this method has been used in some sixteen tentative standards and it is believed that it may have a wider sphere of usefulness, especially in the case of those materials where we may have reason to believe from theoretical grounds that the results obtained by oven drying or by other methods are open to some question.

The method which is described in Method D 95 – 24 in principle involves the distillation of the material with a solvent that is immiscible in water. It was probably first used about 1900 as it is the subject of a German patent in 1901. It begins to appear in the literature in 1903–4–5 and has frequently been erroneously attributed to Marcusson.

TABLE IV.—APPLICATION OF DISTILLATION METHODS FOR MOISTURE.

Asphalt	A. Sommer, *Journal Industrial and Engineering Chemistry*, Vol. 2, p. 181 (1911).
Cellulose	C. G. Schwalbe, *Zeitschrift Angewandte Chemie*, Vol. 21, p. 2311 (1908); Vol. 32, p. 125 (1919).
Coal	P. Schlaffer, *Zeitschrift Angewandte Chemie*, Vol. 27 A, p. 52 (1914). G. A. Brender and Vergen, *Brennstoff Chemie*, Vol. 3, p. 352 (1922).
Creosote Oil	A. Dean, Forest Service, *Circular 134* (1908).
Food Stuffs	Bidwell and Stirling, *Journal Industrial and Engineering Chemistry*, Vol. 17, p. 147 (1925). F. Schaffer, *Mettlel.* Hgg., 6, p. 276. C. Aschman and Arend, *Chem. Ztg.*, Vol. 30, p. 953 (1906). C. E. Gray, Bureau of Animal Industry, *Circular 100.* Thorner, *Zeitschrift Angewandte Chemie*, Vol. 21, pp. 146, 1320 (1908). Rogers, Bureau of Chemistry, *Bulletin Nos. 137–172* (1910). Schaffer and Gurey, Mett. Lebensen, Hg. 7, p. 394. Hoffman, *Zeitschrift Angewandte Chemie*, Vol. 21, p. 2095 (1908). Mai. and Rheinberger, *Z. Nahr. Genussen*, Vol. 24, p. 125. F. Michel, *Chem. Ztg.*, Vol. 37, p. 353 (1913).
Grease	J. Marcusson. *Mitteilungen*, Kgl. Materialprufungsamt, Vol. 22, p. 48 (1904).
Leather	A. Besson, *Collegium*, Vol. 150 (1918).
Lignite	E. Grube, *Braunkohle*, Vol. 58 (1906).
Molasses	G. Testori., *Chem. Zeit.*, Vol. 75 (2), p. 562 (1904).
Mineral Oils	Allen, I. C., Bureau Mines, *Technical Paper No. 25*, 1912. Angli, J., Am. Soc. Quim. Argentina, Vol. VIII, p. 105. Charitschkow, *Chem. Ztg.*, 1906, p. 93; 1908, p. 376. Davin, E., Matt. *Grasses*, 1922, p. 6259. Dean, E. W., *Journal Industrial and Engineering Chemistry*, 1920, p. 486. Dodd, H. V., Calif. Oil Fields, 1923, No. 3, p. 54. Engler, C., "Erdöl, Etc.", 1916, Vol. IV, pp. 6–9. Fuchs, F. C., *Eng. Min. J.*, 1918, Vol. 106, p. 357. Gill, A. H., "Oil Analysis," 1913, p. 22. Graefe, *Petroleum Z.* (1906), Vol. I, p. 813. Hart, R., *Journal Industrial and Engineering Chemistry*, 1918, pp. 598–99. Holde, D., Examination of Hydrocarbon Oils, 1915 (a book). Lieze, *Chem. Ztg.*, 1923, p. 438. Losana, L., *Giorn. Chim. Ind.*, 1922, p. 570. Marcusson, *Mitteilungen*, Kgl. Materialprufungsamt, 1904, p. 48; 1905, p. 58. Marinot, A., *Ann. Chim. Anal. Chim. App.*, 1922, p. 7. Nettel, *Chem. Ztg.*, 1904, p. 867. Norman, W., *Chem. Umchau*, 1923, p. 249. Phillipp, A., *Les Matiers Grasses* 1920, Feb. 15. Pruntzman., Calif. Derrick, 1910, Vol. III, p. 5.

Rakusin, "Untersuchungen des Erdöls," 1906.
Rengade, E., *Comptes Rendus*, 1921, p. 311.
Rodman, C. J., *Journal Industrial and Engineering Chemistry*, 1921, p. 1149.
Rosenthal, *Chem. Ztg.*, 1909, p. 1259.
Schaefer, K., *Chem. Ztg.*, 1924, p. 761.
Wilhelm, V. H., *California Oil Fields*, 1924, No. 9, pp. 5–11.
Wielezinski, *Naphtha*, 1904, p. 25.
" *Petroleum Z.*, 1906, Vol. II, p. 285.
Wilson, R. E., *Journal Industrial and Engineering Chemistry*, 1921, pp. 326–331.
Spices........C. Arrogon, *Schweg. Apoth. Ztg.*, Vol. 53, p. 220 (1915).
A. Scholl and Strohecker, *Z. Nahr. Genussen*, Vol. 32, p. 493 (1916).
H. Lindall, *Journal*, Ass. O. A. Chem., Vol. 2, p. 197 (1917).
Soap.........J. Marcusson, *loc. cit.*
R. Hart, *Journal Industrial and Engineering Chemistry*, Vol. 10, p. 598 (1918).
Tar...........J. W. Jayne, *Journal*, American Chemical Society, Vol. 25, p. 814 (1903).
S. S. Sadtler, *Journal Industrial and Engineering Chemistry*, Vol. 2, p. 66 (1910).
S. R. Church, *Journal Industrial and Engineering Chemistry*, Vol. 3, p. 228 (1911).
W. Spalleholz, *Chem. Weekbld.*, Vol. 15, p. 1546 (1918).

During the last twenty years the method has been applied with more or less success to a very large and varied assortment of materials, a brief survey of which is given in Table IV.

From the results which have been obtained, it would appear that the method has quite a wide sphere of usefulness and appears to possess the following advantages:

1. It is correct in theory;
2. The apparatus required is relatively simple and inexpensive;
3. The time required for a determination is relatively short;
4. Little attention is required in carrying out the method;
5. Large samples of material may be used if desired;
6. Determinations can be duplicated by different laboratories.

The sub-committee does not urge that this method is of universal application and for many materials the committee has found no data as to its practicability, but it believes that the method has considerable promise and that it would be advantageous to extend the purview of the present sub-committee to the end that investigation may be started looking to the application of this method to other types of materials with a view to determining its general suitability, limitations, and accuracy.

The sub-committee therefore recommends that its membership be increased to include representatives of other committees at present having methods for the determination of water to which this new method may appear to be applicable.

Respectfully submitted on behalf of the sub-committee,

W. H. FULWEILER,
Chairman.

APPENDIX I

PROPOSED METHODS OF FLEXURE TESTING OF METALLIC MATERIALS[1]

For metallic materials, flexure tests are secondary in importance to tension tests and compression tests. They are used as control tests for certain brittle cast metals for which flexure test specimens give positive fractures, and which are difficult or expensive to machine to the accuracy of form required of tension or compression specimens.

Flexure specimens of metallic materials, except specimens with a very short span, generally fail by tension in the outer fibers of the convex side; long before failure occurs in a test, Hooke's law ceases to be even approximately true and the accurate computation of stress becomes practically impossible. Moreover, in a flexure specimen the metal of the midportion of depth of the specimen, which is less stressed than the metal at the top and the bottom of the specimen, adds strength to reinforce the extreme fibers. The result of these two conditions is that values for elastic limit and for ultimate strength computed from flexure tests are markedly higher (sometimes 50 per cent higher) than corresponding values from tension or compression tests. Values for elastic limit and for ultimate strength in flexure tests depend also on the form and dimension of the specimen: thus a specimen of circular cross-section shows much higher value for computed stress at elastic limit or rupture than does a specimen of I-section.

Flexure tests are very useful in studying the strength of structural and machine parts of various shapes. It may be pointed out that there is a sharp distinction between the test of the strength of a machine, or of a structural *part*, and the testing of the *material* in that part.

For control tests of the strength of material from which it is difficult or unduly expensive to machine tension or compression specimens, the flexure test has the advantage that, if specimens are made of proper span and supported on properly adjustable supports, secondary stresses which cover up the real strength of the material may be largely avoided. An example of this is found in the testing of cast iron. To get a tensile specimen of cast iron in which the strength will not be seriously affected by localized stress at shoulders or fillets requires careful and expensive machining of specimens. Results, which for control tests are fully as satisfactory as the results of tension tests of carefully machined specimens, can be obtained by the use of an unmachined flexure specimen, the well-known "Arbitration Bar."[2]

I. TEST SPECIMENS

Test Specimens

1. (*a*) Two forms of specimen seem suitable for flexure tests of material: (1) Specimens with a circular cross-section, and (2) Speci-

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[1] Criticisms of these proposed methods are solicited and should be directed to Mr. H. F. Moore, Chairman of Sub-Committee, of Committee E-1, on Mechanical Testing, University of Illinois, Urbana, Ill.

[2] See 1924 Book of A.S.T.M. Standards, p. 391.

mens with rectangular cross-section. At the present time, general dimensions and proportions of flexure specimens can not be given in any detail, but the depth of the specimen (dimension in the plane of the load) should not be greater than one-eighth the span.

(*b*) Flexure specimens now in use are described in the Standard Specifications for Cast-Iron Soil Pipe and Fittings (Serial Designation: A 74) and the Standard Specifications for Cast-Iron Pipe and Special Castings (Serial Designation: A 44) of the American Society for Testing Materials.[1]

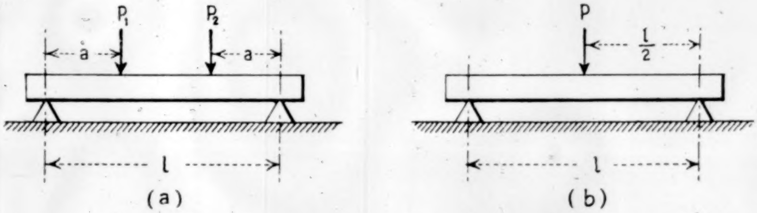

FIG. 1.—Methods of Loading Specimen in Flexure Test.

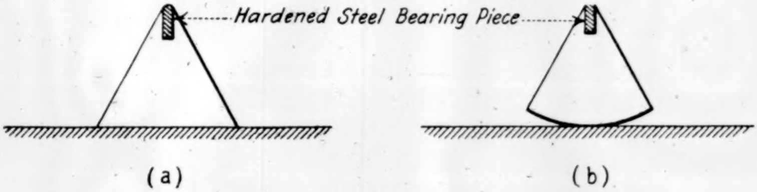

FIG 2.—Knife Edges.

II. LOADING OF SPECIMENS, BEARING BLOCKS

2. In tests of structural parts, such as full-size beams, it is frequently desirable to load the specimen with two symmetrical loads as shown by P_1 and P_2 in Fig. 1 (*a*). The part between P_1 and P_2 is then subjected to a uniform bending moment. For control tests of *material*, however, it is usually satisfactory to use a flexure specimen with a single load at mid-span as shown in Fig. 1 (*b*).

3. It is important that the bearing blocks used in flexure tests should be of such shape that they do not offer appreciable resistance to the bending of the specimen. For most tests of metallic specimens, simple V-shaped blocks fitted with hardened steel bearing plates, such as are shown in Fig. 2 (*a*) are satisfactory. For extreme accuracy, two of the three bearing blocks may be made with a cylindrical base

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[1] 1924 Book of A.S.T.M. Standards.

so that they can rock as the extreme fiber of the convex side of the specimen stretches. Fig. 2 (*b*) shows such blocks. For accurate testing of flexure specimens of rectangular cross-section, it is desirable that two of the three bearing blocks shall be adjustable for twist present in the specimen, so as to avoid introducing twisting stress during the flexure test. Such blocks are shown in Fig. 3. This adjustment to avoid twist is not necessary for specimens of circular cross-section.

4. In flexure tests of metallic materials, bearing strips should be placed between the specimen and the edge of the bearing blocks. The width of such strips should be at least as great as that of the specimen, their length should not be less than one-third their width and their thickness should not be less than one-quarter the depth of the specimen.

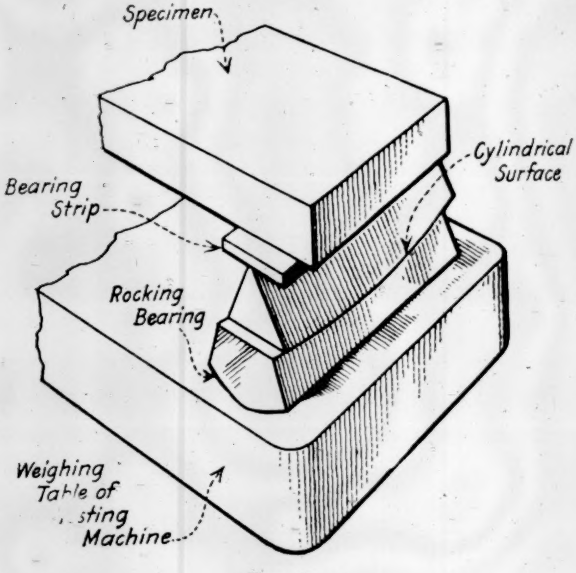


FIG. 3.

III. MEASUREMENT OF DEFLECTION

5. In flexure tests of metallic materials it is frequently desired to measure the deflection of the specimen at rupture. The amount of this deflection is considered to be an inverse measure of the brittleness of the material. In flexure tests the measurement of deflection can be made roughly by measuring the motion of the head of the testing machine, but such a measurement includes not only the

deflection of the specimen, but also the depth of indentation of bearing blocks and of specimen into the bearing strips. This depth of indentation is greater under the central load than at the end supports of the specimen, and the measurement of deflection by use of a device such as that shown in Fig. 4 eliminates the measurement of this largest indentation. The device shown in Fig. 5 is a still more accurate device for measuring deflection. It is important that the deflection-measuring device be such that it is not injured when the specimen breaks. For general use in testing flexure specimens of metallic materials, a deflection measuring device which can indicate a deflection of 0.001 in. is sufficiently sensitive.

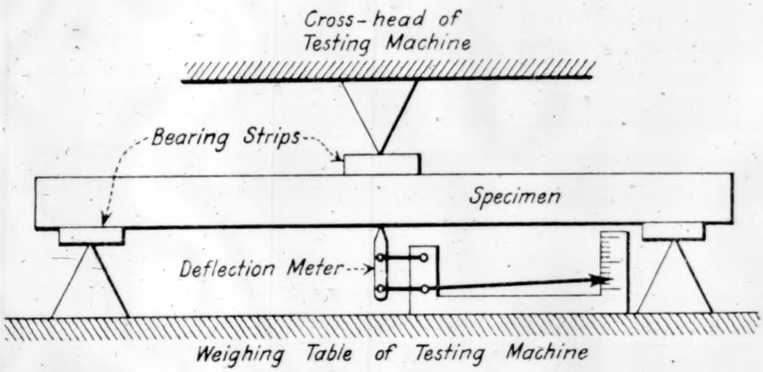
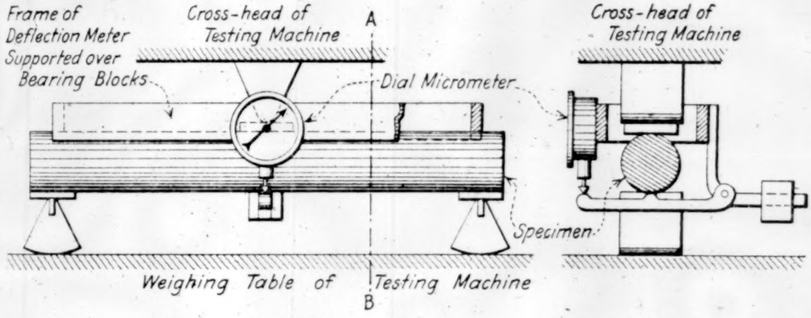

Cross-head of Testing Machine
Bearing Strips
Specimen
Deflection Meter
Weighing Table of Testing Machine

FIG. 4.—Apparatus for Measuring Deflection.


Frame of Deflection Meter Supported over Bearing Blocks
Cross-head of Testing Machine
A
Dial Micrometer
Cross-head of Testing Machine
Specimen
Weighing Table of Testing Machine
B

Elevation.

Section A-B.

FIG. 5—Apparatus for Measuring Deflection.

IV. SPEED OF OPERATION OF TESTING MACHINES

6. In flexure tests, the allowable speed of testing machine is markedly affected by the cross-section and the span of the specimen.

For all flexure tests to determine elastic limit and for flexure tests to destruction of specimens of brittle materials (including hardened steel), the speed of the machine, by which is meant the speed of the cross-head when the machine is running idle, shall not be greater than 0.2 in. per minute.

V. DETERMINATION OF ELASTIC LIMIT, YIELD POINT AND MAXIMUM FLEXURAL LOAD

7. (*a*) In general, elastic limit values for ductile metals can be determined more satisfactorily from tension tests or compression tests than from flexure tests. The elastic limit for a material like tempered spring steel, which is very difficult to machine, is sometimes determined by flexure tests of rectangular specimens. The elastic limit of a metal determined by a flexure test has, in general, a higher value than the elastic limit of the same material determined by a tension test, and in comparing elastic limits this fact should be borne in mind.

(*b*) In determining the elastic limit of a material by a flexure test, it is usually most convenient to use a load-deflection diagram. For determining the load at the elastic limit in a flexure test either Method II, as given in Section 17 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 – 24 T) of the American Society for Testing Materials,[1] or a still more precise method seems suitable. From the load at the elastic limit, the stress in the extreme fibers of the specimen can be calculated from the standard formulas of mechanics of materials. For specimens loaded with one load, P, at the middle of the span of length l, these formulas for the stress, S, become

$$S = \frac{2.55\ Pl}{d^3}, \text{ for a specimen with circular cross-section of diameter } d;$$

$$S = \frac{3}{2}\frac{Pl}{bh^2}, \text{ for a specimen with rectangular cross-section of depth } h \text{ and width } b \text{ (load vertical).}$$

8. Owing to the reinforcement of the outer layers of a flexure specimen by the less-stressed inner layers, it is doubtful whether the yield point can be accurately determined in a flexure test.

9. No detailed discussion seems necessary of methods of determining the maximum load carried by a specimen in a flexure test, except to give a warning against reporting a value of maximum load for a specimen which bends without breaking to the limit determined by the motion possible under the condition of test.

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[1] See Tentative Methods of Tension Testing of Metallic Materials (E 8-25 T), p. 854.—ED.

10. The results of flexure tests to failure are sometimes calculated by using the formula for elastic flexure and from the load at rupture and the dimensions of the specimen calculating a value measured in pounds per square inch (kilograms per square centimeter) and called the "modulus of rupture." However, the flexural action is by no means purely elastic up to the point of rupture, and the value of the modulus of rupture not only is not a true stress, but it is not independent of the form and dimension of the specimen. For flexure tests of metallic materials, the use of the modulus of rupture is of doubtful value and it seems a better plan to follow the practice of specifying in detail the form, dimensions, and methods of loading of the specimen, and then to report the test results in terms of load at rupture for such a specimen.

VI. PLOTTING STRESS-STRAIN DIAGRAMS

11. The discussion and directions given in Section 22 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 - 24 T) apply to plotting stress-strain diagrams (load - deflection diagrams) for flexure tests.

APPENDIX II.

PROPOSED METHODS FOR TORSION TESTS TO DETERMINE THE MECHANICAL PROPERTIES OF METALLIC MATERIALS UNDER SHEARING STRESS[1]

In many machine parts and structural members shearing stresses are of primary importance. This is true for parts subjected to torsion and for parts, such as rivets, subjected to transverse forces spaced over a short span.

Whenever a part (for example, a test specimen) is subjected to axial tension (or axial compression) there is set up on any plane inclined to the axis a shearing stress, and on the planes inclined at 45 deg. to the axis this shearing stress is a maximum, and is equal to one-half the axial tensile (or compressive) stress.

Whenever a part (for example a test specimen) is subjected to pure shearing action perpendicular to its axis, as in the case of a shaft under pure torsion, there is set up a tensile stress or a compressive stress on any plane inclined to the axis, and considering two planes perpendicular to each other and inclined at 45 deg. to the axis there is set up on one a tensile stress equal in magnitude to the maximum shearing stress; on the other there is set up a compressive stress equal in magnitude to the maximum shearing stress.

The statements made in the two foregoing paragraphs are demonstrated in the standard texts on the mechanics of materials, and they are given to show the importance of considering the shearing resistance of materials as well as the tensile resistance and the compressive resistance.

The ratio of shearing resistance to tensile resistance for metallic materials varies from about 0.50 (probably slightly greater than 0.50) for the elastic limit of ductile metals to a value greater than 1.00 for the strength at fracture for brittle metals.

For metallic materials the ratio of *stiffness* in shear to stiffness in tension (or compression) is less than unity for all metals studied. This relative stiffness is measured by the ratio of modulus of elasticity in shear to modulus of elasticity in tension, and for wrought metals this ratio averages about 0.4.

I. TEST SPECIMENS

Test Specimens.

1. For metallic materials whose shearing resistance is greater than the tensile resistance no satisfactory form of test specimen has yet been devised. In any shearing test speciman of such metal failure will occur by tension along a plane inclined to the planes of maximum shearing stress. This is illustrated in Figs. 1 (*a*) and (*b*). In Fig. 1 (*a*) is shown a torsion specimen of ductile metal for which the shearing strength is less than the tensile strength. Failure has occurred on a plane perpendicular to the axis.

NOTE.—A shearing stress of equal magnitude was developed along radial planes parallel to the axis.

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[1] Criticisms of these proposed methods are solicited and should be directed to Mr. H. F. Moore, Chairman of Sub-Committee, of Committee E-1, on Mechanical Testing, University of Illinois, Urbana, Ill.

In Fig. 1 (*b*) is shown a torsion specimen of a brittle metal (cast iron). Failure has occurred by tension across a plane inclined to the axis of the specimen. Possibly the shearing strength of brittle metals can be measured by the use of short compression specimens, but this is a matter requiring further investigation before a conclusion can be reached.

2. Shearing tests of specimens of ductile metals are sometimes made by the use of a testing rig similar to that shown in Fig. 2. For such tests, the test specimen is merely a short piece of rod of the metal tested. Such a test may be of some value as a control test, but the results of such a test depend to a considerable degree on the hardness and the sharpness of the edges of the hardened steel plugs or plates which bear on the specimen. In such a test, the distribution of stress across the cross-section of the specimen is unknown; the stress directly under the cutting edges of the plugs must be decidedly higher than the stress at the opposite side of the specimen.

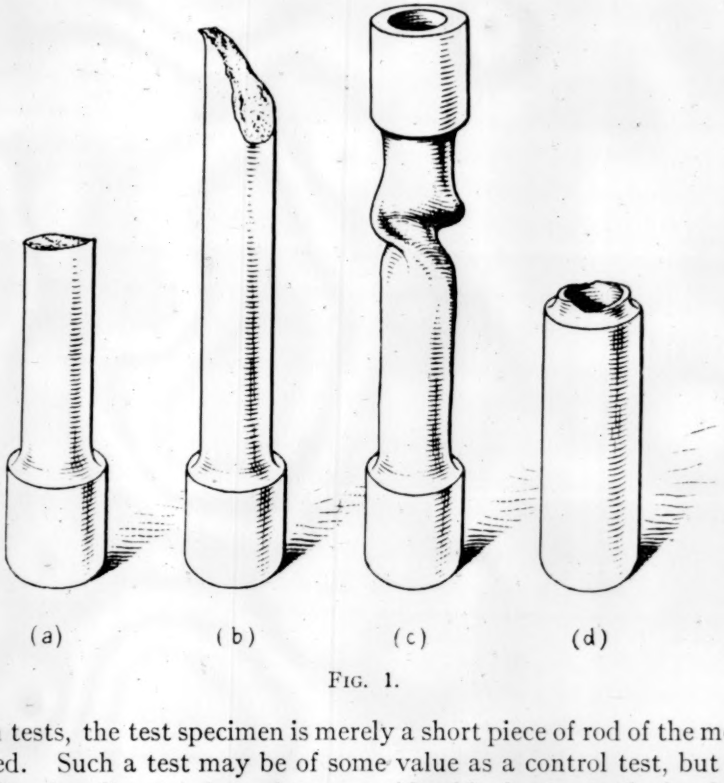

FIG. 1.

3. Torsion tests seem to be the most suitable tests to use for determining the shearing resistance and shearing stiffness for metals whose shearing resistance is less than their tensile resistance. To

secure approximately even distribution of shearing stress over the cross-section of the specimen, thin-walled hollow circular specimens are recommended. Fig. 3 shows a form of hollow torsion specimen suitable for determining the elastic limit in shear for ductile metals. This specimen is not suitable for determining shearing strength (maximum shearing stress carried by specimen in a torsion test, analogous to tensile strength, see Section 11) because final failuer occurs not by shear but by collapse along an inclined axis, as is illustrated in Fig. 1 (*c*). The walls of the hollow specimen should not be thinner than one-tenth of the outside diameter of the specimen, else the tendency to fail by compressive collapse in a direction inclined to the axis will appreciably affect the value determined for the elastic limit.

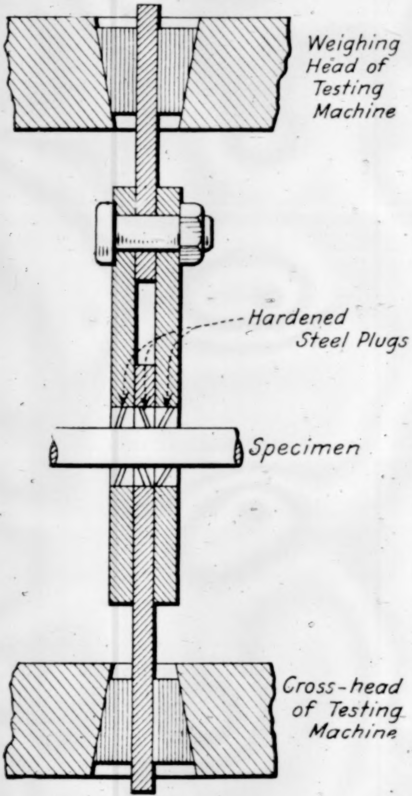
Weighing Head of Testing Machine
Hardened Steel Plugs
Specimen
Cross-head of Testing Machine

FIG. 2.

4. For determining the shearing strength (stress corresponding to maximum load carried during torsion test to destruction, see Section 11) the hollow specimen shown in Fig. 4 is recommended.

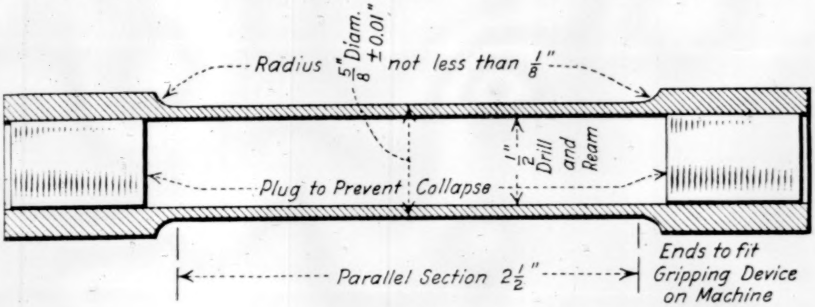
Radius not less than 1/8"
5/8" Diam. ± 0.01"
1/2" Drill and Ream
Plug to Prevent Collapse
Parallel Section 2 1/2"
Ends to fit Gripping Device on Machine

FIG. 3.

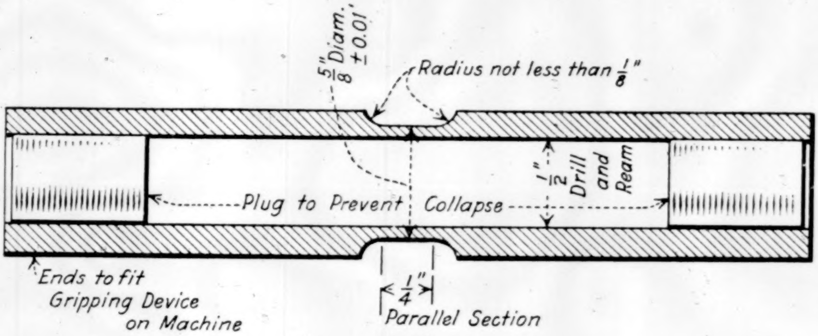
5/8" Diam. ± 0.01"
Radius not less than 1/8"
1/2" Drill and Ream
Plug to Prevent Collapse
Ends to fit Gripping Device on Machine
1/4"
Parallel Section

FIG. 4.

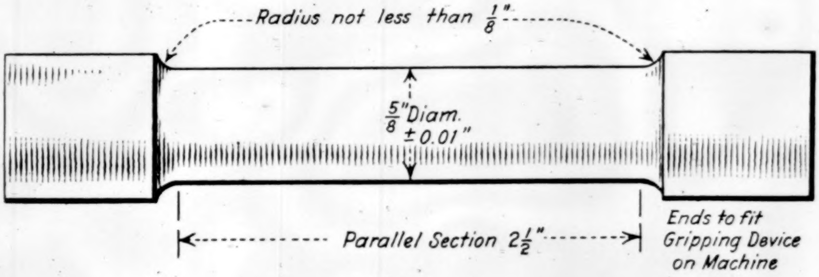
Radius not less than 1/8"
5/8" Diam. ± 0.01"
Parallel Section 2 1/2"
Ends to fit Gripping Device on Machine

FIG. 5.

The length of the turned-down portion of this specimen is made short so that collapsing action on an inclined plane is not an appreciable factor in causing failure. Fig. 1 (*d*) shows a typical shearing failure of this type of specimen.

5. For control tests of ductile metals to determine elastic limit in shear, solid specimens may be used. The values given by tests of solid specimens are, in general, greater for solid specimens than for hollow specimens. This is due, partly at least, to the reinforcing action of the understressed fibers in the central part of the specimen. A suitable solid specimen for control tests to determine elastic limit in shear is shown in Fig. 5. Values for elastic limit in shear obtained for one metal from tests of solid specimens should not be compared with values for another metal obtained from tests of hollow specimens.

6. For determining the modulus of elasticity in shear for a metallic material a solid specimen may be used similar to that shown in Fig. 5 except that the length of parallel portion between fillets should be at least 9 in.

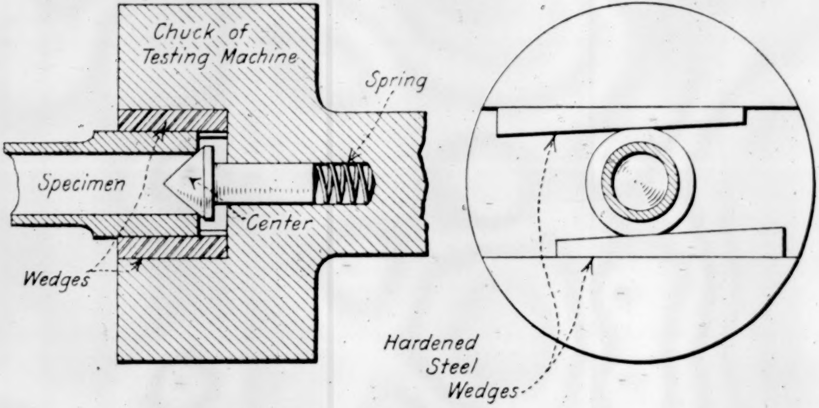

FIG. 6.

II. GRIPPING DEVICES FOR TORSION SPECIMENS

7. For soft metals and metals of medium hardness, self-centering torsion grips consisting of three or four pivoted jaws acting on the principle of the pipe wrench are generally satisfactory. The gripping device shown in Fig. 6 is suitable for general use in torsion testing and is especially suitable for torsion tests of hard metals.

III. SPEED OF OPERATION OF TESTING MACHINE

8. In making torsion tests of specimens of metallic materials, the speed of the machine, by which is meant the speed of the moving chuck when the machine is running idle, or the actual speed of operation of a hand-operated machine, shall not be greater than the following values:

For tests to determine elastic limit in shear	0.10 r.p.m. per inch of gage length
For tests to determine shearing strength	0.20 r.p.m. per inch of gage length

IV. DETERMINATION OF ELASTIC LIMIT AND YIELD POINT IN SHEAR, AND SHEARING STRENGTH

9. (*a*) The term "elastic limit" is defined as follows:

Elastic Limit.—The greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress.

With the exception of Method I the same general methods for determining the elastic limit in shear shall be used as are used for determining the elastic limit in tension, as given in Section 17 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 – 24 T) of the American Society for Testing Materials.[1] A strainometer to be suitable for use in determining the elastic limit in shear by means of a torsion test should be capable of indicating a twist of 0.01 deg. per inch of gage length on specimen.

(*b*) The elastic limit in shear should be given in terms of stress, and a torsion testing machine gives values in terms of twisting moment. For any value of twisting moment, T, measured in inch-pounds, outside diameter of specimen, D, measured in inches, and inside diameter of specimen d, measured in inches, the shearing stress S_s on the outer fibers of a torsion specimen is:

$$S_s = \frac{5.1\ TD}{D^4 - d^4} \qquad (1)$$

For a solid specimen, d becomes zero and

$$S_s = \frac{5.1\ T}{D^3} \qquad (2)$$

10. The term "yield point" is defined as follows:

Yield Point.—The stress in a material at which there occurs a marked increase in strain without increase in stress.

For determining the yield point in shear, Method I given in Section 18 of the Tentative Methods of Tension Testing (E 8 – 24 T) shall be used. The stress at the yield point should be calculated from the twisting moment by the use of the formulas given in Section 9 (*b*).

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[1] See Tentative Methods of Tension Testing of Metallic Materials (E 8 - 25 T), p. 854.—Ed.

11. The term "shearing strength" is defined as follows:

Shearing Strength.—The maximum shearing stress which a material is capable of developing.

NOTE.—In practice it is considered to be the maximum stress carried by a specimen representing the material in a torsion test to failure, under definite prescribed conditions.

For a hollow specimen, the shearing strength is calculated from the maximum twisting moment in a torsion test by the use of Eq. 1, given in Section 9 (*b*). For a solid specimen, the formula cannot be used because above the elastic limit the stress is not uniformly distributed over the cross-section of the specimen. No detailed discussion of the method of determining maximum load is necessary, except to give a warning against reporting a value for shearing strength for a torsion specimen which fails by diagonal collapse rather than by shear.

V. PLOTTING STRESS-STRAIN DIAGRAMS

12. The discussion and directions given in Section 22 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 – 24 T) apply to plotting stress-strain diagrams for torsion tests.

DISCUSSION

Mr. Comstock.

Mr. G. F. Comstock[1] (*presented in written form*).—The method for determining elastic limit presented as information by Committee E-1 on Methods of Testing,[2] is a logical and satisfactory method, a great improvement over the methods previously advocated by the committee. It seems now in order to inquire why the committee should propose three methods, each determining a truly different property, and yet designate the results obtained from the three methods by the single term "elastic limit." Method I,[3] as has been pointed out in previous discussions, gives hardly more than a sort of refined yield point; the proposed method gives the proportional limit; and Method II gives the so-called "Johnson limit" which lies somewhere between the other two points on the stress-strain curve. The action of Committee E-1 in authorizing the use of the term "elastic limit" for the results of all three methods leads to a serious state of confusion in regard to the real meaning of the term. The "elastic limit" in one paper, for instance, may be an entirely different thing from what is called the "elastic limit" in another paper. Committee E-1, or Committee E-8 on Nomenclature and Definitions, should clear up this situation by taking some definite stand on the real meaning of "elastic limit."

The note to the definition of "elastic limit," given earlier in the report,[4] admitting that the proportional and elastic limits are customarily accepted as equivalent, is very good, but that note is not consistent with the statement that Methods I and II give the elastic limit. The authors of the very valuable papers that have appeared recently in the Proceedings on the properties of various steels have evidently appreciated this situation, and have shown clearly that the yield point, Johnson limit, and proportional limit are quite different. Yet Committee E-1 proposes that Method I, giving a sort of yield point, and the Johnson method both determine the elastic limit, while also stating that practically the elastic limit is equivalent to the proportional limit!

In the report of the committee, the excellent advice is given to have "one clear-cut general method instead of numerous descriptions

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[1] Consulting Metallurgical Engineer and Metallographist, Niagara Falls, N. Y.

[2] See p. 409.

[3] Tentative Methods of Tension Testing of Metallic Materials, see p. 854.

[4] See p. 403.

Mr. Comstock. for a certain determination." The proposed method for determining "elastic limit" is indeed a clear-cut method for the determination of the proportional limit, and if we are to assume that the elastic limit is practically determinable in the same way, this method should stand alone as the standard. We have other terms for the results obtained by Methods I and II, and the position of the committee would be far more logical if it were stipulated that the term "elastic limit" should not be applied to the results of those methods.

I have one other criticism of this report, and that is the definition of "shearing strength" appearing in the proposed methods of torsion testing. It seems to me that the note which follows the definition is better than the definition itself.

Mr. Woodward. MR. R. W. WOODWARD[1] (*presented in written form*).—Committee E-1 presents in its report, as information, a "Proposed Additional Method for Determining Elastic Limit." In the letter ballot of the committee the writer voted negatively on this proposal, not as an objection to the method but to the title of the method.

This method appears to be clearly a method for the determination of proportional limit as defined earlier in the report.[2] Although it is true that there is considerable loose use of the terms "elastic limit" and "proportional limit," it is the writer's observation that the preferred term for the property determined as the departure of a stress-strain curve from a straight line is "proportional limit." For example, all reports from the Bureau of Standards are so construed; also at this morning's session each speaker referred to this determination as the "proportional limit" and it is not recalled that "elastic limit" was once mentioned.

The writer believes that to label this method a determination of "elastic limit" would be a step backward and that the sub-committee should give consideration to the common usage of these terms.

Mr. Moore. MR. H. F. MOORE[3] (*Chairman of Sub-Committee on Mechanical Testing, of Committee E-1 on Methods of Testing*).—The arguments that have been advanced against the recognition of Methods I and II for determining elastic limit seem to be consistent arguments from the viewpoint of the testing engineer who thinks of elastic limit or proportional limit *as some value determined by a certain definite method.* To the sub-committee this viewpoint seems less fundamental than the viewpoint of elastic limit as the *limiting stress at which appreciable inelastic action begins.* Taking this second viewpoint it is seen that

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[1] Chief Metallurgist, Whitney Manufacturing Co., Hartford, Conn.

[2] See p. 403.

[3] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

Mr. Moore.

for different materials different methods may be necessary in determining the elastic limit. In determining by chemical analysis the percentage of an ingredient present in a material different methods are allowed under different conditions; under some conditions quick, approximate methods are allowed, under other conditions precise, time-consuming methods are required. So for some metals the range of stress from first appreciable inelastic action to stretch visible to the unaided eye is a narrow range, and Method I, a quick, rather rough method, is allowable for determining elastic limit. In other metals where this range of stress from first appreciable inelastic action to marked strecth is a broad range, Method I would not be satisfactory and Method II or a still more precise method (possibly the proposed Method) will be required.

It should never be forgotten that it is not at all sure that there is any absolute elastic limit or absolute proportional limit for any material.

The sub-committee will keep this discussion under advisement, and I hope that others who hold a different viewpoint on this question from that held by the sub-committee will continue to present their viewpoint.

REPORT OF COMMITTEE E-4

ON

METALLOGRAPHY

Committee E-4 has held three meetings during the year; during the Annual Meeting at Atlantic City, June 24, 1924, a second at Washington, D. C., October 23, 1924, and the third in Philadelphia, on March 19, 1925.

Sub-Committee I on Selection and Preparation of Samples is active under the temporary chairmanship of Mr. G. F. Comstock, who is taking the place of Mr. Wm. Campbell, during the latter's absence abroad.

Mr. E. H. Dix, Jr., of the Aluminum Co. of America is working on a report as to the preparation of samples, etching, and determination of the constituents in aluminum alloys, which report he expects to have ready for the annual meeting.

The sub-committee is also considering the "Pulsifier Method" of preparation of samples for microscopic examination and photography, and if the committee reports favorably, the matter of incorporating the method in the Standard Methods of Metallographic Testing of Iron and Steel (E 3 – 24) will be considered at the next meeting.

Sub-Committee II on Nomenclature and Definitions, Mr. A. Phillips, chairman, reports that the only matter for consideration is a satisfactory definition for metallography.

Sub-Committee III on Thermal Analysis, Mr. W. E. Ruder, Chairman, has prepared a proposed Recommended Practice for Thermal Analysis of Steel which is recommended for acceptance for publication as tentative, as appended hereto.[1] This report is the culmination of a lot of work having been carried out by several laboratories for quite some time.

The report of Sub-Committee V on Micro-Hardness, Mr. H. S. Rawdon, Chairman, is appended hereto.

Sub-Committee VI on X-ray Crystal Analysis, Mr. Zay Jeffries, Chairman, has prepared an extremely interesting report, which is appended hereto.

As to work planned for the future, reports or papers are in view on "High Magnification and its Value in Metallography" and "Best Recommended Practice for High Power Photomicrography," also "On Means to Avoid Eye Strain in Connection with Microscopic Work."

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[1] See p. 890.—Ed.

This report has been submitted to letter ballot of the committee, which consists of 48 members, of whom 40 have voted affirmatively, none negatively, and 8 have refrained from voting.

Respectfully submitted on behalf of the committee,

H. C. BOYNTON,
Chairman.

G. F. COMSTOCK,
Secretary.

EDITORIAL NOTE

The proposed Recommended Practice for Thermal Analysis of Steel was accepted for publication as tentative and appears on page 890.

REPORT OF SUB-COMMITTEE V ON MICRO-HARDNESS

The sub-committee on micro-hardness has been carrying out for the past two years a series of hardness tests to obtain information as to the usefulness of the scratch hardness method and the reproducibility of the results obtained by this method. A series of specimens was prepared by the U. S. Bureau of Standards and this set of "unknowns" was sent to each cooperating member in turn so that in this manner variations resulting from differences in material were minimized since the members used the same specimens successively. Upon completion of the test by a member, the results and specimens were returned to the chairman of the sub-committee and the specimens were repolished before they were sent on to the next observer.

TABLE I.—MATERIALS TESTED FOR HARDNESS.

SPECIMEN	MATERIAL
No. 1	Cobalt.
No. 2	Iron, electrolytic, remelted.
No. 3	Lead.
No. 4	Copper, electrolytic, remelted.
No. 5	Antimony.
No. 6	Nickel, electrolytic, remelted.
No. 7	Copper-tin alloy.
No. 8	Tin.
No. 9	Tin-antimony alloy (hard crystals used throughout).
No. 10	Copper-phosphorus alloy.
No. 18	0.85-per-cent carbon steel, hardened by quenching from 750° C. in water.

The materials used, which were referred to by number only in all correspondence, are listed in Table I. All determinations were made with the same type of scratch-hardness instrument, a commercially available form being used. No instructions or recommendations were sent out by the sub-committee concerning its use.

Table II summarizes the results obtained by seven observers working independently. Observers A, B, and C are members of the same laboratory and used the same instrument, hence it may be concluded that instrumental errors may be disregarded for this set of results. Observers D, E, F, and G are in widely separated laboratories and there is every reason to believe that the work was carried out in each absolutely independently of any of the others.

The wide variations in the results obtained by different observers for the same specimen may be attributed, in part, to the lack of experience in the use of the method. It is believed that the results clearly show the need for a standard method of procedure in carrying out the scratch hardness test in addition to that of using the same type of instrument. The sub-committee does not believe that any sweeping conclusions should be drawn on the basis of the above results, at present, and is arranging an additional series of tests which, it is planned, will be based upon two different metals in widely differing conditions of hardness as indicated by other methods of hardness measurements.

TABLE II.—HARDNESS RESULTS BY SCRATCH METHOD.

Specimens Arranged in Order of Hardness.

Specimen	Material	Average Width of Scratch, microns						
		Observer A	Observer B	Observer C	Observer D	Observer E	Observer F	Observer G
No. 18	Steel	1.2	...	1.6	...	2.1	2.3	2.0
No. 7	Copper-Tin	3.1	3.0	2.7	...	2.5	2.8	3.7
No. 1	Cobalt	3.8	3.3	3.2	11.4	3.2	4.6	4.2
No. 10	Copper-Phosphorus	4.0	3.9	2.7	...	6.2	3.2	3.1
No. 2	Iron	6.8	6.0	3.3	12.1	5.3	3.5	6.3
No. 9	Tin-Antimony	7.4	7.4	4.1	...	7.4	3.9	8.3
No. 6	Nickel	6.7	5.7	3.4	9.0	9.3	8.3	7.0
No. 5	Antimony	6.2	8.2	6.1	...	4.8	7.8	8.1
No. 4	Copper	10.4	10.4	7.9	9.2	6.3	9.4	6.9
No. 8	Tin	25.8	31.3	23.1	...	17.6	15.4	27.4
No. 3	Lead	38.7	...	34.0	24.7	13.0	12.2	40.7

Respectfully submitted on behalf of the sub-committee,

H. S. RAWDON,
Chairman.

REPORT OF SUB-COMMITTEE VI ON X-RAY METALLOGRAPHY

X-rays have been used to a limited extent to determine the location of cavities, cracks and certain other structural variations in castings or other pieces of metal. This field is referred to as Metal Radiography.

X-rays have been used quite extensively during the last few years to determine the arrangement of atoms or molecules in crystals, size of grains or particles in crystallin aggregates and the orientation of such crystallin material with respect to the external boundaries of a sample. This study may be called X-ray Crystallography. X-ray Crystallography as applied to metals and Metal Radiography comprise what may be called X-ray Metallography. It is the purpose of this report to discuss briefly the methods employed and some of the results obtained in X-ray Metallography.

Metal Radiography

1. *Introduction.*—There are two important applications of metal radiography in the routine tests of the metallurgical laboratory of the foundry. The first of these is in inspection tests to determine whether or not a casting is fit for use. In this service the X-ray does not replace other tests, but is used to reveal possible defects not revealed by other tests. The second of these is in foundry control tests. In this service X-ray pictures reveal internal defects in pilot castings, that are corrected by changes in foundry technique.

Beside the application to routine tests, radiography is valuable as a method of investigation in foundry research. Some use has already been made of it in this field, and the possibilities revealed are encouraging.

2. *Physical Basis.*—Metal radiography makes use of the fact that exceedingly short wave-length radiation will penetrate matter opaque to light. X-rays used for this purpose have wave lengths of from 0.1 to 0.05×10^{-8} cm., approximately 100,000 times shorter than the wave length of yellow light, which is around 5890×10^{-8} cm.

In passing through metals or other dense media, X-radiation is progressively absorbed with greater depth of penetration, according to a logarithmic law. The absorption also depends on the atomic weight of the material penetrated, being greater for larger atomic weights. In metals where the radiation encounters cracks, cavities, sand and slag inclusions, it is absorbed to a different extent than the radiation passing through adjacent sound metal, so that a photographic film placed to intercept the emergent X-ray beam shows, on development, regions of varying density or definitely outlined images corresponding to the defects in the interior of the metal. The films with these markings constitute the X-ray record of the defects in the metal. It is then necessary to interpret the negatives.

3. *Apparatus and Methods.*—The apparatus involved includes essentially a high-voltage X-ray tube with necessary auxiliary equipment for obtaining and controlling the high potentials, X-ray intensifying screens, and dark room equipment. The tube is usually mounted in a lead-lined room or is otherwise provided with lead screens to protect the operator from injurious exposure to the X-rays. In addition to the X-ray tube and its accessories, the laboratory must have facilities for handling heavy castings. At present the most practical tube is the 200-kilovolt, 8-milliampere air-cooled Coolidge tube used by the medical profession for deep therapy treatments. It has been the practice at Watertown Arsenal to operate this tube for intermittent exposures at potentials from 220 to 250 kilovolt and 5 milliamperes. Such use above rated potential probably shortens the life of the tube, but used in this way the life of the tube has been found to be about 200 hours, which will mean in ordinary practice a tube replacement about every ten months. The intensifying screens for metal thicknesses above 2 in. consist of sheets of calcium tungstate. Double screens are used. The photographic film, which is coated on both sides, is placed between the two screens. For steel sections less than 2 in. in thickness, metal screens may be used. For sections of less than $\frac{3}{4}$ in., intensifying screens are not needed. Where screens are used, the image on the negative is formed by the direct action of the X-rays plus the effect of the screen fluorescence. The screen fluorescence consists of secondary radiation given off by the material of the screen under the influence of the primary radiation from the target of the X-ray tube. Calcium tungstate gives a blue-green fluorescence that may account for 95 per cent of the negative density. Metal screens are much less efficient, but have the advantages of producing finer grained images and of shielding the films from some of the secondary radiation from the specimen being radiographed.

The metal usually employed is lead in the form of foil about 0.002 in. thick.

The secondary radiation that is made to serve a useful purpose in the intensifying screens is given off also by every object in the path of the X-ray beam and the stray radiation thus produced coming from sources other than the target of the tube tends to fog the films. To protect against it lead is interposed to absorb all radiation except that coming from the target.

If a casting is to be studied it is first examined to determine the position in which it was cast, the location of gates and risers, probable location of chill rods, variation in section, etc. It is then marked off into areas through which pictures are to be taken. These areas are selected as the ones likely to show defects. Regions subjected to great stresses or of unusual importance from other considerations are also included. Usually from 40 to 60 per cent of the total area of the casting is covered in this way, the amount depending on the object of the examination. The areas selected are numbered with paint and lead numerals are placed over the painted ones. The lead numbers appear in the negatives and serve as indicators to insure that the section was actually penetrated and later serve as marks for orienting the finished negatives with the casting. The casting is then placed under the X-ray tube, the film is fixed in position beneath a particular area with the tube above it, and the exposure made. After all the pictures are obtained the casting is re-examined and compared with the films and the defects are charted on the surface of the casting.

4. *Problems and Results.*—Results achieved, so far as they relate to foundry problems, have been for the most part obtained in the testing laboratories of Watertown Arsenal where an X-ray section was installed in the fall of 1922 for metallurgical research and radiographic testing. Due to the fact that this was a pioneer laboratory the first problem of importance was the development of a suitable laboratory technique. In this study it was found that the thickness of metal that could be successfully radiographed was for practical purposes a linear function of the voltage across the tube up to 200,000 volts, the current through the tube and the exposure time being constant. The practical limit of thickness that could be radiographed was found in the case of steel to be about 3 in., in which thickness a cavity $\frac{1}{16}$ in. in diameter could be detected. Pictures through 3 in. were obtained with 30 minutes' exposure. The maximum thickness of steel radiographed was 4 in., the pictures being obtained with seven hours and forty minutes' exposure. Since that time, the practical limit has been raised to $3\frac{1}{2}$ in. thickness of section for favorably shaped steel castings

and a picture through 4.1 in. of solid steel has been obtained with one hour and thirty minutes' exposure.

The next problem that presented itself was the problem of diagnosing films. It was necessary to find out what the spots on the films meant. To get at the information required, castings found by the X-ray tests to be defective were cut up and revealed defects were compared with their images in the negatives. This work was enlarged to include a systematic study of casting defects. As a result of the investigation it was found possible to diagnose the defects found in the film negatives with a fair degree of certainty. In many cases the defects could be correlated with definite causes.

In foundry control testing the radiographic method has been used successfully to develop proper casting technique in the Watertown Arsenal and in some commercial foundries. It is probable that this application will be the most important one for metal radiography. This use forces definite study of specific defects with consequent specific education of foundry personnel. This study has resulted in general improvement in casting quality and in a reduction in the percentage of rejected castings.

In the field of inspection testing, the method has been employed successfully both in this country and abroad. Much ordnance material such as aëroplane parts, fuses for shells, and similar small pieces were examined in this way in England during the war. Carbon electrodes, built up mica, turbine blades and various other articles of manufacture have been inspected by the radiographic method in this country but the more spectacular applications in the past two years have been in the inspection of heavy castings. An illustrative example of this use is the examination of the turbine shell, steam line, and control valves of a 1200 lb. per sq. in. steam pressure installation in a modern superpower electric plant. In this case, the value of the service into which the castings entered was such that the X-ray tests were regarded as necessary insurance. In these tests some castings were rejected which would have passed any ordinary tests. It is probable that radiography will find an increasing application in inspection tests where, as in this case, the loss from breakdown would greatly exceed the cost of the tests.

5. *Suggestions and Limitations.*—Further development in X-ray apparatus is needed. Higher voltage tubes should be developed. There is probably a limit beyond which higher voltages would not be advantageous, but that limit has not yet been reached. A million-volt tube should be able to test sections 10 in. thick.

The greatest present limitation to a more extensive use of radiographic tests is the cost and this is due partly to the lack of flexibility of the apparatus. X-ray pictures cost at present from one dollar to five dollars apiece and it takes sometimes forty pictures to cover an important casting. It is probable that radiography will always be expensive as a method of inspection. Its economical application to foundry control testing seems possible, but here also its value will depend on the individual problems of the foundry. With the present rapid evolution of the mechanical appliances of industry there is an ever growing demand on structural units for increased strength and dependability; metal radiography should be helpful in this connection.

Anyone interested in a more detailed study of metal radiography will find some helpful references at the end of this report.

X-RAY CRYSTALLOGRAPHY

1. *Physical Basis:*

Two structural states of matter are now well recognized, namely, crystallin and amorphous. Nearly all solid metals are essentially crystallin. Crystallinity depends upon an orderly arrangement of the molecules of a substance. In the molten condition the molecules are arranged in a haphazard manner. Liquid metals are typical examples of the amorphous state. When a metal crystallizes, the atoms group themselves according to definite and repeating patterns. Metals are monatomic, that is, their molecules contain only one atom so the atom becomes the unit in the crystal pattern. The pattern which forms the foundation upon which a crystal is built is known as a "space lattice." It consists of a series of points in space such that every point is situated similarly to every other point. A space lattice may be imagined as divided into cells by three sets of parallel planes. The planes of each set are at equal distances from each other and parallel; the distance between planes may be different in the various systems or in different materials, and the three sets of planes may form any angles with each other. The points of intersection of these planes constitute the space lattice. These planes determine by their intersections the crystallographic axes of a crystal. The directions of these axes with reference to some line or plane determine the orientation of a crystal.

In a single crystal of metal the atoms in the whole volume must be arranged according to the same space lattice and the orientation of the crystallographic axes must be uniform. A perfect single crystal may be referred to as a group of molecules in which the entire space occupied by the group can be divided into congruent parallelopipeds, each containing the same least number of atoms arranged in precisely the same way.

It was formerly considered that in order to be classed as a single crystal the surface must be bounded by plane faces called crystal faces. The present tendency is to use the term crystal with reference to the arrangement of the molecules and to disregard external shape. A crystal, however, may be bounded by plane surfaces and this is usually the case where it has formed slowly in a liquid of lower melting point. In most metals, however, the boundaries of the crystals (grains) are irregular and have no known relation to their space lattices. This is no hindrance in X-ray crystallography which gives information concerning the volume arrangement rather than the surface arrangement of atoms.

Particles which are of dimensions comparable with the wave length of visible light scatter or diffract such light in all directions, the spatial distribution of the scattered light depending upon the size and other properties of the particles. Colloid particles seen under the ultramicroscope present good examples of such scattering.

Single atoms are of dimensions comparable with the wave length of X-rays, and accordingly scatter or diffract such X-rays in all directions. The method of applying X-rays in the study of crystallography is to expose a representative sample of matter to a suitable beam of X-rays, to find the spatial distribution of the diffracted beams, and to deduce the relative positions of the atoms or molecules in the sample.

In the case of metals a representative sample may contain a single crystal, an aggregate of grains of one constituent either with random or more or less special orientation with respect to some line or plane or both, or an aggregate of different constituents with variations in type, size and orientation of the crystals. In crystallin aggregates a limiting case is reached when the individual units are so small that their structure and orientations cannot be determined with certainty.

If a sample consisting of only two nearby atoms diffracted X-rays from a single source to the same point on a screen not too near the diffracting atoms, there would in general be a difference between the distances traversed by the X-rays from the source to the screen along the two possible paths. If this difference happened to be zero, or any other integral multiple of the wave length of the X-rays, the two diffracted beams would produce their maximum possible effect at the selected point on the screen. If the difference in path were half a wave length, or any other odd integral multiple of half a wave length, the effect would be a minimum. By studying the pattern of maxima and minima on the screen, it would be a simple matter with known positions of source, sample and screen, to find the orientation of the line joining the centers of the diffracting atoms, and if the wave length of the X-rays were given, the distance between these centers would also be perfectly determinate.

Exactly the same reasoning applies to the diffraction pattern due to any number of atoms, and a complete knowledge of the pattern would serve to determine the arrangement, in space, of their centers. If the arrangement were not extremely simple, however, the diffraction pattern would be too complicated to be interpreted, and if many atoms were distributed quite at random the resultant effect would be like that of a single atom of great scattering power, and all evidence that separate atoms were present would disappear. Even in a monatomic liquid the case is a little better than this because there is between atom-centers in such a liquid a definite minimum separation, and there is, therefore, a maximum of intensity in the diffraction pattern corresponding to the great number of pairs of atom-centers, which are at any moment separated by almost exactly this distance. In liquids with more complicated molecules there is still more detail in the diffraction pattern.

In crystals, at least in crystals like those of most metals, wherein the atoms do not wander far from their equilibrium positions, the diffraction patterns become fairly simple, because atoms located at the points of a space-lattice can give diffracted X-rays agreeing in phase at only a few points on a screen, and can only do this if the incident X-ray beam contains a particular wave length for each such possible point. A single crystal exposed to X-rays having a wide range of wave lengths gives in this way a diffraction pattern of separated spots, each spot being due to rays of a different wave length. This spot pattern is often referred to as a Laue pattern in honor of O. von Laue, the discoverer of the effect. W. L. Bragg pointed out that each spot may be considered as due to specular reflection from some particular family of planes determined by points of the space-lattice. This concept is extremely convenient if care is taken to remember that "reflection" from such a family of planes can only occur for a specified wave length at a given angle of incidence.

If there is more than one atom in each of the parallelopipeds mentioned earlier, congruent space-lattices may be associated with each of them and the X-rays diffracted by the atoms at the points of such space-lattices will differ in phase by different amounts at the various spots of their common diffraction pattern. If the atoms on the different space-lattices have different scattering powers this also affects the intensities of the spots. A study of the relative intensities of the spots of the Laue pattern therefore affords data for fixing the number, relative positions and scattering powers of the several space-lattices, that is, for fixing the number, relative positions and chemical identity of the adjacent atoms at any point in the crystal. Such analysis is seldom necessary in metallography except in the case of intermetallic compounds.

It is often convenient to choose as the unit of structure a parallelopiped or other simple figure which contains two, four or six atoms of the same sort rather than one only. Such a unit of structure is preferred when the symmetry of the crystal is high, and is then so chosen that it possesses the highest possible symmetry. Three examples of frequent occurrence may be mentioned.

Pure metals which form crystals belonging to the cubic system may be divided into two classes. The convenient unit of structure is for both classes a cube, but the number of atoms in the unit cube is different for the two. In one class half of the atoms are placed at the corners of the cubes forming the space-lattice and half at their centers. This space-lattice and unit of structure are both described as body-centered cubic and there are two atoms per unit cube. In the other class one fourth of the atoms are placed at the corners of the cubes, and three fourths at the centers of the three sets of opposite faces. This space-lattice and unit of structure are described as face-centered cubic and there are four atoms per unit cube.

Metals which form crystals belonging to the hexagonal system may be treated by taking as the unit of structure a right rhombic prism formed of two right prisms with bases isosceles triangles. Half the atoms are placed at the corners of the prisms and half at the centers of one of the two sets of triangular prisms, differently oriented, into which the whole crystal can be divided. Even this unit does not well exhibit the symmetry of the crystal, and the hexagonal prism of which the rhombic prism forms a third is often used in describing the structure. This larger unit contains six atoms. The space-lattice and unit of structure are described as close-packed hexagonal, because, for a particular ratio of prism height, c, to hexagon edge, a, *viz.*, for $c/a = \frac{2}{3}\sqrt{6} = 1.633$, this is one of the two possible closest packings of spheres. The other closest packing is the face-centered cubic arrangement and the two are equally compact. It may be mentioned that the close-packed hexagonal space-lattice is not a fundamental type and will not be found in treatises on mathematical crystallography.

The diffraction pattern will change as the crystal is rotated about any axis. If the axis chosen is perpendicular to the incident beam of

X-rays and is one of the principal axes of the crystal, some of the spots will move on the screen along the trace of a plane perpendicular to the axis of rotation. This experimental arrangement is the basis of all X-ray spectrometry, for, with a given crystal, X-rays of a specified wave length will be diffracted only at certain azimuths of the crystal. The pattern of diffracted beams obtained during rotation is the spectrum of the incident X-rays. Conversely, using X-rays of known wave length, such rotation of a single crystal permits accurate measurement of the distances between important planes of its space-lattice. This method, first used by W. L. Bragg, is principally useful for crystals of rather simple structure and is the one most suitable for detecting the diffracted beams by their ionizing effects.

An aggregate of crystals of any material, oriented at random, is equivalent in scattering X-rays to a single crystal of the same material given in succession all possible orientations. It should therefore, on a plane perpendicular to the incident beam, give a pattern with central symmetry. Such a pattern, if due to X-rays having a wide range of wave lengths, is too complicated to be read, and the simplest way of reducing its complexity is to use as nearly as possible monochromatic X-rays, that is, X-rays of a single wave length. This prevents most of the crystals from giving any diffracted beams at all, and analysis shows that those still capable of doing so must give a diffraction pattern consisting of the traces on the screen of a series of circular cones with common vertex at the sample. The vertex angles of these cones depend upon the wave length of the X-rays and upon the characteristics of the space-lattice. The intensity of the X-rays diffracted along any cone depends, as in the more general case, upon the arrangement of the atoms within the unit of crystal structure, and upon the angle of scattering. It also depends upon the number of sets of planes giving the same angle of scattering.

An assemblage of crystals not oriented at random will give a pattern intermediate in character between that due to a single crystal and that due to a chaotic aggregate of crystals. The curves in which the cones of the latter pattern cut the screen break up into more or less sharply defined maxima which become more and more like the spots of the Laue pattern or Bragg pattern as the preferences for a particular orientation become more pronounced. The limiting pattern will, of course, be that of a single crystal rotated successively through all the positions consistent with the preferred orientation. The pattern depends upon the relative orientation of the incident beam and the axis or plane with respect to which the crystals are preferentially oriented. The preferred orientation and its relation to the unique axis or plane in the sample can therefore be derived from a single pattern suitably obtained.

The details of the geometrical analysis of diffraction patterns (crystallograms) are beyond the scope of this report. The reader is referred for such details to the leading books and papers on this subject, all of which are of recent date.

2. *Apparatus and Methods:*

In this country the usual practice has been to use high-power, high-vacuum X-ray tubes delivering electron currents of 10 milliamperes or more, and to reduce the necessary precision of measurements upon the diffraction patterns by placing the photographic plate or film at a considerable distance from the sample. The target in the X-ray tube is preferably at the same distance from the sample as is the screen, and with such dimensions no slits are necessary to limit the direct beam of X-rays. Wide slits are always used, however, to reduce the amount of scattered radiation reaching the sample. In Europe the effort has been directed to reducing the time of exposure and the requisite power plant for the high-voltage current. This has led to the use of metal X-ray tubes with short distances between target and sample and between sample and photographic film, and to the selection of target materials giving characteristic X-rays of great wave length, not capable of high energy dissipation. Metal X-ray tubes require continuous pumping and are not adapted to the highest vacua, so they are not self-rectifying even when the target (anode) is water-cooled. The use of such tubes requires, therefore, rather more manipulative skill than the use of permanently sealed glass tubes with hot-wire cathodes and they are less suitable for routine metallography.

The voltage to be used across the X-ray tube depends upon the sort of X-rays desired. For Laue patterns it is essential to have a fairly wide range of wave lengths without sharp variations in intensity with wave length. This is best effected by making the target a metal, tungsten, which has no characteristic X-rays when run at voltages below about 70,000, and running the tube at a peak voltage of, say, 60,000. For patterns with monochromatic X-rays the most convenient source is a tube with a water-cooled molybdenum target, run at such a voltage (30,000) that its characteristic K-series X-rays are strongly developed. It is usually advisable to cut out part of the general radiation and characteristic X-rays of shorter wave length than the $K\alpha$ lines of molybdenum by filtering the X-rays through zirconia either near the source or just before reaching the photographic film. Zirconia forms an especially efficient filter in this case because the element zirconium has its K-series absorption limit between the $K\alpha$ and $K\beta$ lines in the X-ray spectrum of molybdenum. The excess absorption on the short wave length side of this limit greatly reduces the intensity of the $K\beta$ and $K\gamma$ lines, with respect to the intensity of the $K\alpha$ lines. The general radiation of short wave length can be reduced, with a sacrifice of speed, by reducing the voltage on the tube so that the maximum intensity of the general radiation has about the same wave length as the K-lines of the target. The $K\beta$ lines of molybdenum are not eliminated by this means and may interfere with the analysis of the patterns in critical cases.

For examinations in which the X-rays must pass through the sample before affecting the photographic film, the shape and dimensions of the sample should be so chosen that roughly two-thirds of the incident radiation is absorbed or scattered in passing through it. Table I, hitherto unpublished, gives appropriate thicknesses for various elements when using a molybdenum target X-ray tube.

TABLE I.—SUITABLE DIMENSIONS FOR CRYSTAL ANALYSIS OF VARIOUS ELEMENTS USING THE USUAL MOLYBDENUM X-RAYS.

ATOMIC NUMBER	ELEMENT	OPTIMUM THICKNESS OF SHEET SAMPLE, MILS	ATOMIC NUMBER	ELEMENT	OPTIMUM THICKNESS OF SHEET SAMPLE, MILS
4	Be	720	40	Zr	2.5
6	C	240	42	Mo	1.7
11	Na	125	47	Ag	1.5
13	Al	31	50	Sn	2.1
20	Ca	16	56	Ba	3.0
26	Fe	1.6	73	Ta	0.34
28	Ni	1.2	74	W	0.26
29	Cu	1.1	79	Au	0.20
30	Zn	1.3	82	Pb	0.30

If the thickness of material placed in the X-ray beam is much greater than that given in Table I, it absorbs too much of its own diffracted radiation, and if the thickness is much less, the stray diffraction from other objects becomes very troublesome in the longer times of exposure which are then necessary. For cases in which the diffracted rays emerge from the sample on the side exposed to the incident X-rays it is advisable to use much thicker samples.

In case the crystals are not sufficiently small, the diffracted beam from each properly oriented crystal is so intense that the pattern due to a stationary sample consists of separately visible spots or dashes, the shape of the spot depending, as in the Laue pattern, upon the appearance of the source as seen from the sample. This affords useful additional information in some cases, but if accurate measurements are important the pattern is best rendered as smooth as possible by rotating the sample or by rocking it through a suitable angle. The average intensity of the pattern is not much changed by such progressive changes in its orientation, because the number of crystals brought into action for short periods increases as the time of action of each crystal is reduced.

In studying assemblages of crystals it is often convenient to reduce the grain size by grinding, filing or the like, and when such powdered samples of low absorptive power are being examined, the sharpness of the pattern can be improved by limiting the diffracted beams as well as, or instead of, the incident beam. When the crystals are too small the maxima become broad and diffuse and only those deflected through small angles can be detected. In the limit the pattern is indecipherable. Annealing powdered metal improves the definition of the resulting patterns and is advisable, unless annealing obliterates the characteristic differences which are under investigation.

While it is possible to observe X-ray diffraction patterns on a fluorescent screen, and while this is convenient in seeking particular orientations in single crystals or in very thin films, the general practice is to photograph the patterns for measurement. Laue patterns are usually of moderate angular extent and are always taken on plates suitable for measurement of rectangular coordinates. Patterns showing preferred orientations in crystal assemblages are also studied on plates. In both these types of pattern the best results require an approximately circular beam of incident X-rays. Patterns obtained from chaotic assemblages of crystals are more suitably received on strips of film pressed against a cylindrical frame with the sample on the axis of the cylinder. The source is so placed with respect to the sample that the focal spot from which the X-rays radiate appears as a narrow line parallel to the axis of the cylinder. One or both sides of the pattern may be photographed, the latter permitting somewhat higher accuracy in results for the same precision in placing the sample.

The accuracy of measurement is also increased by using an elongated sample with its length parallel to the slits (or to the plane of the focal spot if slits are not relied upon). The cones of diffracted beams from the crystals on the illuminated edge of the sample have, in this arrangement, a plane for part of their envelope, and the trace of this plane on the screen is a straight line on which accurate settings of a measuring instrument are particularly easy.

The use of comparison patterns is quite general and has a number of advantages. It allows correction for the non-uniform shrinkage of photographic film and is of particular value where the geometry of the arrangement is variable or in calibrating any new set-up. If it is relied upon, however, to correct considerable errors in the position of the sample, the resulting appearance of accuracy is illusory, especially in metallographic work where it is impossible to mix the standard substance with the substance to be investigated. It should be noted that when mixture is possible the absorptions of the investigated material and of the standard substance should be adjusted to approximate equality by diluting the former, since the position of diffraction maxima depends slightly upon absorption in the sample.

In what have been called rotation diagrams of single crystals, *i. e.*, Bragg spectra plus spectra not in the plane of incidence, the incident beam is limited by slits parallel to the axis of rotation and plates are generally used in preference to films because of the higher accuracy they permit. Cylindrical films, however, give a pattern on which approximate analysis is easier, the lateral spectra being parallel to the central spectrum.

The measurement of diffraction patterns varies in precision depending upon the purpose of the work. Simple inspection serves to identify the more common sorts of crystal structure and to disclose preferred orientations of such crystals. More complicated cases can be solved with fair accuracy by the use of transparent charts laid over the photographs, or by specially ruled scales applied to them. The highest precision requires the microphotometric location of maxima in spectrometric work and the measurement of angles to single seconds of arc. Metal crystals are rarely of such perfection as to deserve very refined measurement of their diffraction patterns, but in the comparison of closely similar structures the fourth figure in the numerical results may be significant if the work is of high quality. The use of ionization methods, with the increase of resolving power made possible by the use of very narrow slits, and of multiple slits in parallel has so far not been justified in metallography.

For examination of metals at temperatures much higher than atmospheric somewhat elaborate schemes for electrical heating and protection of specimens from oxidation, are required. It is customary to house the sample under X-ray examination in a gas-tight container which comprises both the film holder and the specimen holder.

3. *Results:*

The application of X-ray crystallography to the study of metals and alloys has made available information of considerable value to workers in the field of physical metallurgy and metallography. In fact, before the first utilization of this method of study there were some problems which were in a state of arrested progress on account of the inadequacy of all the known methods of attack. For a few of these problems the X-rays have already brought a sufficient volume of new knowledge to give a great impetus toward adequate solutions.

The study of atomic spacing and arrangement in metals by X-rays carries with it the possibility of obtaining certain other information, and for these other closely related data the metallographist is as greatly indebted to the X-ray methods as he is for the simple atomic arrangement data.

The subjects, familiar to metallographists, which have been illuminated by X-ray crystal analysis may fairly well be divided into two groups. In a first class may be put those studies which reveal chiefly the atomic arrangement itself in the many or few similar grains under study, and in a second class the studies which involve the grains as units, and which yield data upon their orientation, number or extent.

In the first class of studies would be included identification of and discrimination between constituents; determination of allotropy, mechanism of solid solution formation, coring, coefficient of thermal expansion and stress-strain relations. In the second class would be put studies on grain size effects and establishment of preferred orientation by extreme cold work or by electrodeposition.

In this report some of the important utilizations of this new method of study will be considered separately with appropriate examples of each application.

(*a*) *Identification of Constituents.*—The X-ray pattern of a suitable fine-grained sample of an element or compound becomes essentially a kind of qualitative test for its presence. There are, however, pairs of elements producing patterns which are of the same type and with almost identical spacings between the lines. The patterns of tungsten and molybdenum, for example, are difficult to distinguish and the patterns of gold and silver are only slightly less so. However, the first work for the X-rays was the establishment of the atomic arrangement—lattice type and spacing—in the various metals. Hull and Davey have done much of the best of this work, and, with others, have explored practically the entire field of metals. Most metals, fortunately, crystallize in very simple arrangements.

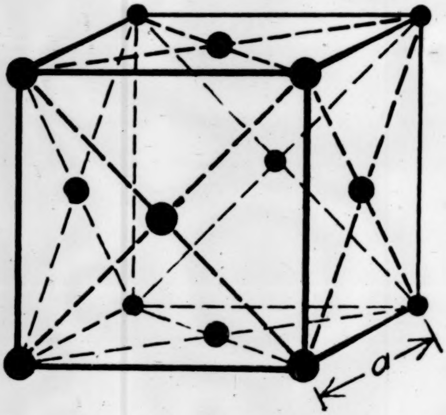


FIG. 1.—Face-Centered Cubic Crystal Structure.

Metal	Parameter, *a*, Ångstrom Units	Metal	Parameter, *a* Ångstrom Units
Al	4.05	Pd	3.950
Ca	5.56	Ag	4.060
Fe (γ)	3.60	Ce	5.12
Co	3.554	Ir	3.805
Ni	3.50	Pt	3.930
Cu	3.60	Au	4.08
Rh	3.820	Pb	4.92
		Th	5.04

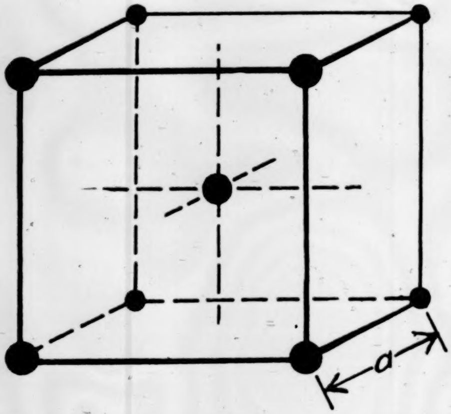


FIG. 2.—Body-Centered Cubic Crystal Structure.

Metal	Parameter, *a*, Ångstrom Units	Metal	Parameter, *a*, Ångstrom Units
Li	3.50	Fe (α)	2.86
Na	4.30	Mo	3.143
V	3.04	Ta	3.272
Cr	2.895	W	3.150
K (−150° C.)	5.20		

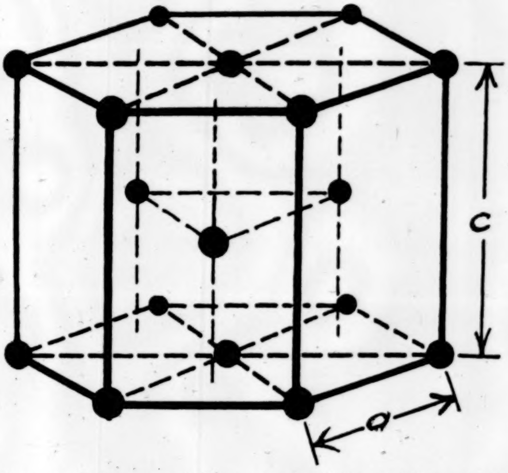


FIG. 3.—Hexagonal Close-Packed Crystal Structure.

Metal	Parameter, a, Ångstrom Units	$C = \frac{c}{a}$	Metal	Parameter, a, Ångstrom Units	$C = \frac{c}{a}$
Mg	3.22	1.624	Ru	2.686	1.59
Ti	2.97	1.59	Cd	2.960	1.89
Co	2.514	1.633	Ce	3.65	1.62
Zn	2.67	1.86	Os	2.714	1.59
Zr	3.23	1.59	Be	2.283	1.58

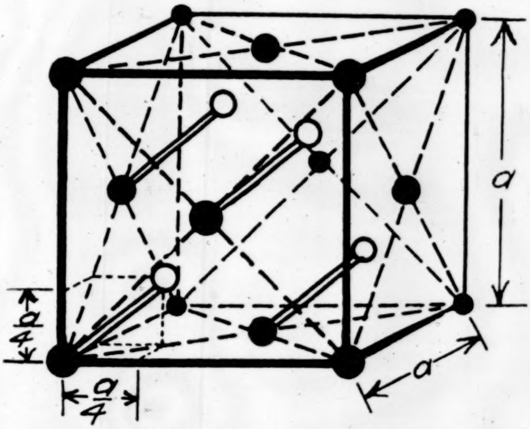


FIG. 4.—Tetrahedral Cubic Crystal Structure.

Tetrahedral cubic. It may also be shown for comparison on a hexagonal lattice in which the vertical axis is the trigonal axis of the cube. Actually, the arrangement consists of two interpenetrating, face-centered lattices somewhat removed from the positions that would result in the simple cubic arrangement. The two systems of atoms are shown connected by double lines, point for point.

Metal	Parameter, a, Ångstrom Units
Diamond	3.55
Silicon	5.42
Tin (gray)	6.46
Germanium	5.61

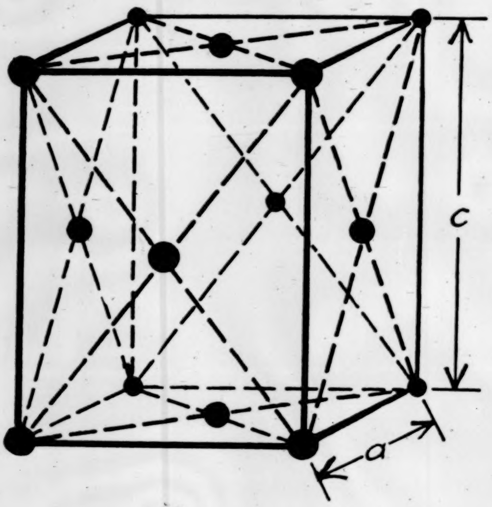


FIG. 5.—Face-Centered Tetragonal Crystal Structure.

Metal	Parameter, a, Ångstrom Units	$C = \frac{c}{a}$
Indium	4.58	1.06

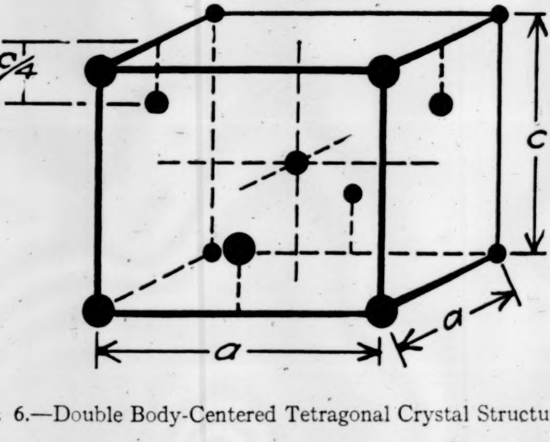
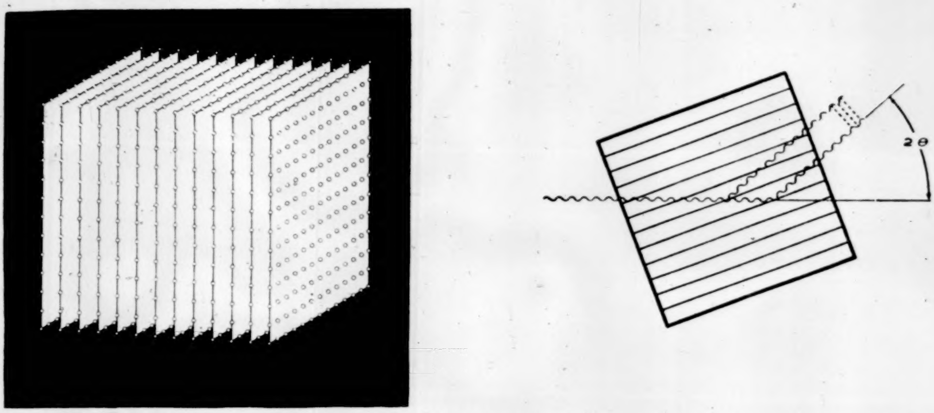
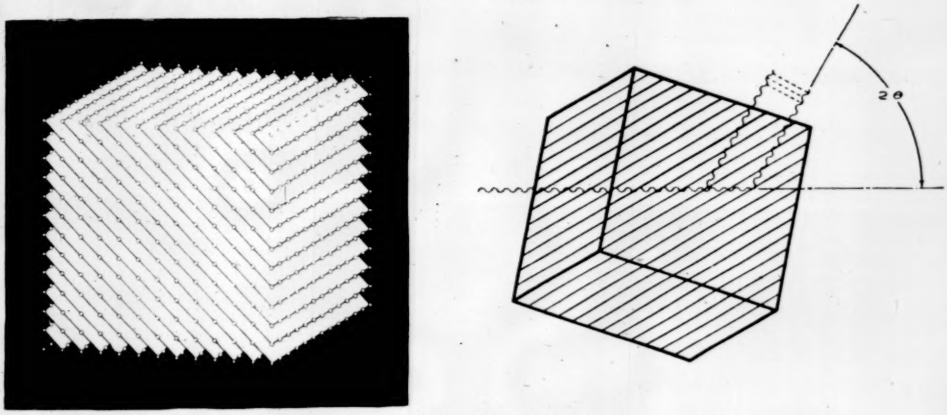


FIG. 6.—Double Body-Centered Tetragonal Crystal Structure.

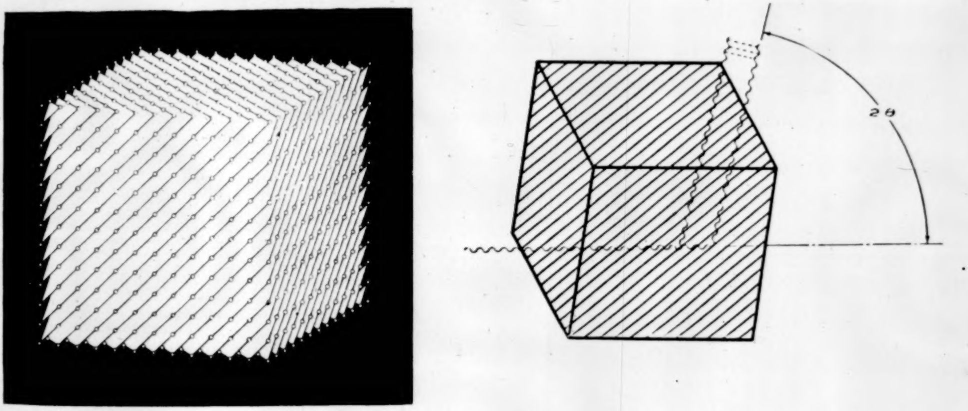
Metal	Parameter, a, Ångstrom Units	$C = \frac{c}{a}$
Tin (white)	4.85	0.65



(a) (100) Plane.



(b) (110) Plane.



(c) (111) Plane.

FIG. 7.—Illustrates X-ray Reflection from Those Three Families of Planes of a Simple Cubic Space-Lattice Which Have the Three Greatest Inter-Planar Distances. These are the (100), (110) and (111) planes, respectively.

The reflection angles are drawn correctly for the X-rays corresponding to the molybdenum Kα line and a simple cubic space-lattice with cube edge 1 Ångstrom unit.

The accompanying diagrams (Figs. 1 to 6) show the various types of space-lattice found in pure metals, together with the principal metals crystallizing with each lattice. The parameters are given in Ångstrom units (10^{-8} cm.)

In a series of ordinary alloys ranging from pure A to pure B, the X-rays may be employed to show the exact number of crystal types involved (at the temperature of examination) regardless of the nature of the constitution diagram. Thus in the copper-zinc series, there are but four actual constituents to be found. These four may be identified as the alpha solid solution, the beta solid solution, a compound of rhombohedral crystal structure, and a solid solution of a slight amount of copper in zinc. Once these constituents have been identified by their patterns, changes of structure such as those produced by heat treatment, can be followed by the appearance, disappearance or change in relative intensity of the component patterns.

Similarly, the presence of precipitated constituents can be determined by the X-rays, sometimes even before the particles are visible under the microscope. The solution and reprecipitation of the complex carbide in high-speed steel is easily followed by X-ray crystallography.

The retention of austenite (gamma iron solid solution) in some quenched steels is most clearly shown by the superposition of its pattern upon the lines of alpha iron characteristic of iron and of most hardened steels. The persistence of such austenite can be determined by reheating samples until they no longer yield the austenite lines in the pattern.

(*b*) *Allotropy.*—X-ray analysis appears to be inherently the ideal method of study for the determination of allotropic changes. Unless one chooses to regard as allotropic such changes as are evidenced by the magnetic changes in iron at 768° C. and in nickel at 358° C. the essence of allotropy appears to be atomic rearrangement. This method of study has been applied to cobalt, iron, tin and carbon, and the precise shifts in the arrangement of the atoms are well known.

Cooling iron, when pure, changes at about 900° C. (and at lower temperatures when containing carbon, nickel or manganese) from a face-centered cubic (gamma) form to a body-centered cubic (alpha) form. Actually, iron, if fairly pure, freezes from the melt in a crystallin arrangement identical with the low temperature or alpha form but at about 1400° C. changes, as it cools, to the face-centered cubic or gamma form. The extremely high temperature form, even though it may be identical with alpha iron, is termed delta iron.

Iron containing certain elements in solid solution may be preserved in the gamma condition at room temperature after an extremely rapid cooling from a temperature at which that form is stable.

Cobalt may exist at room temperature either in the face-centered cubic form (like nickel and gamma iron) or in the close-packed hexagonal form (like zinc and magnesium).

Diamond (like silicon) is a complicated isometric (cubic) crystal involving the interpenetration of two face-centered cubic lattices of carbon atoms in a manner to produce tetrahedral characteristics.

Graphite may be regarded as having for its atomic arrangement an interpenetration of two congruent close-packed hexagonal lattices of carbon atoms. There is some doubt, however, as to the exact relative position of the two lattices and it may be that two different structures exist.

(*c*) *Solid Solutions.*—The nature of solid solution has been studied extensively by means of X-ray crystallography and some significant data obtained. When one metal dissolves in another metal as solid solvent the atomic arrangement remains unchanged in type up to saturation. The average atomic spacing, however, does change with composition. In this case the solute atoms appear to adapt themselves to the habits of the solvent atoms, and when they become too numerous to conform they set up an entirely different arrangement—a new phase.

It has further been shown that a truly continuous series of solutions from pure A to pure B can only exist between elements of the same crystallin type. In pairs such as copper-manganese and nickel-chromium in which continuous solid solution series have been assumed on account of the smoothness of freezing point curves, two different solid solutions are, over some range of compositions, invariably found in a single specimen. This indicates the overlapping of the solubility of A in B and of B in A. A very real meaning is hereby given to the words "solvent" and "solute" in such solid solutions.

It often happens that the solubility of A in B increases with rise in temperature. If a solid solution of the two is just saturated with respect to A at a high temperature, a slow cooling will precipitate the excess, while if the solid solution is cooled with great rapidity it may be preserved, highly supersaturated, at room temperature. Reheating at some lower temperature will then induce precipitation. In some important cases the precipitating excess is in the form of an intermetallic compound A_xB_y. Such processes may in some cases be followed by X-ray methods. In the case of the solution of carbon in iron at high temperature and the precipitation of iron carbide at lower temperatures it is found that for some reason iron carbide, Fe_3C, in the form of very minute particles, yields a pattern too weak to permit following its changes with accuracy. The presence of the tungsten atom, however, in the complex carbide of high-speed steel renders its pattern relatively much stronger.

In most cases it is likely that solid solution is accomplished by the substitution in the lattice of atoms of solute metal for atoms of solvent. The general tendency for perfection of crystallinity to persist in solid solutions and the tendency toward shrinkage of the lattice argue for this mechanism. It is possible, however, that carbon dissolves in iron by occupying the interstices between atoms.

(*d*) *Coring.*—Coring is the phenomenon exhibited by solid solutions when they solidify in grains which have non-uniform composition. The first crystallites to freeze are usually rich in the high-melting constituent and the last material to freeze around these dendritic skeletons is therefore poor in the high-melting element. The grain-boundaries in such ingots, caused by the final junction of one growing nucleus with its neighbors, are normal in appearance. Within these grain-boundaries are, however, regions of very different compositions. In these regions of different composition there is found the disparity in atomic spacing characteristic of different proportions in the series of solid solutions.

X-ray patterns of such cored structures show broad diffuse lines which are practically obliterated at the larger angles of reflection. The pattern is analogous to that which would be obtained from a mixture of powdered alloys with a range of composition. It is very likely that there may be more than one orientation in primary grains of this character. The effect of this gradation of lattice constants in a cored solution produces quite a complicated pattern, and the evidence obtained from such patterns promises to shed much light upon the mechanism of solidification in solid solutions.

Prolonged annealing, especially if accompanied or preceded by some working, restores ingots of such solid solution alloys to homogeneity and a state of much more nearly perfect crystallinity with uniform atomic spacing.

(*e*) *Grain-Size Effects.*—As has been explained, smooth continuous bands in the patterns produced from assemblages of crystals (Hull or Debye-Scherrer patterns) require the cooperation of an enormous number of differently oriented crystals. In fabricated metals this condition often obtains. With a fewer number of grains in the volume exposed to the X-rays (that is, with larger grain size) the lines are not smooth but dashed, for then the separate diffraction images of the source are not sufficiently numerous to give, by overlapping, a smooth band. We have at hand then a definite and sensitive indication of the grain size in every crystallogram. With very coarse-grained metal it is possible to obtain not even one strong diffraction band on the film. Some weak bands, due to imperfect monochromatism of the X-rays, will usually show. At the other extreme of fragment size, the patterns give evidence equally characteristic. When the crystal fragments are in the colloidal range each band is diffuse, since the single particles are not of sufficient size to produce a sharp diffraction image. It is in particles wherein there are only a few hundred atom layers that this effect is noticeable—a condition rarely met with in solid metals.

The effect of grain size upon the appearance of the crystallogram has been utilized to reveal the mechanism of grain fragmentation by cold work. When very coarse-grained bars (which yield one or two heavy dashes for a pattern) are deformed by any of the ordinary methods, such as forging, rolling or wire drawing, the resulting material develops a more and more complete pattern of continuous bands as the deformation increases up to a certain amount. This proves the development of new orientations which were absent in the original few grains. The results prove rotation of grain fragments as well as the translation which was held by some investigators, prior to this demonstration, to be a sufficient cause for the subdivision of the original grains.

Another example of grain-size effects is afforded by the investigation of beta brass. When large grains were produced of a proper size to yield, if of simple structure, a complete pattern of the dashed type, the sharpness and clearness of these diffraction spots indicated the presence of a true single-phase and not, as had been suggested, a eutectoid material. The dashes were such as would be associated with a material having a true grain size corresponding to the apparent grain size as viewed at low magnification, and no other lines appeared on the film to indicate a second constituent.

A very important application of X-rays to metals, in which the estimation of grain size is important, is in the examination of martensite. When steel is heated to a sufficiently high temperature for a long time, uniform grains of austenite are formed which grow to a size visible to the naked eye. If a pattern were made of such material (at the high temperature) it would be that of gamma (face-centered cubic) iron, with the dashed appearance already described. When such steel is quenched in water a very hard structure results which has been named martensite. At least one school of metallurgists has regarded this hard form of iron as a special allotrope. Under the microscope it shows quite clearly the boundaries of the large austenite grains. But X-rays yield the pattern of alpha iron and, what is more important, the grains appear to be very small. The austenitic grain boundaries are therefore no longer the only grain boundaries. Each grain of austenite has been transformed into many grains of ferrite.

(*f*) *Establishment of Preferred Orientation.*—Not only does severe cold work break up large crystals into smaller ones, but when applied to extend the metal repeatedly in the same manner, it tends to arrange these small crystals in certain preferred orientations, depending upon the crystal structure and upon the type of cold work. Thus it has been found in the case of tungsten that severely rolled sheet and severely drawn wire have the greater portion of the fine crystals of which they are composed arranged with their crystal axes in definite positions with respect to the working direction. Effects of a similar kind have been observed in molybdenum, iron, copper, silver and aluminum.

Preferred orientation is likewise found in certain metals when deposited electrolytically. In this case the axis with respect to which the arrangement is symmetrical is normal to the plated surface. The occurrence of these effects is closely connected with the state of strain in electro-deposits. Samples of tungsten and molybdenum worked to produce preferred orientation of the crystals showed similar preferred orientation after recrystallization. Different results in the face-centered cubic metals, silver and copper, have been reported. As would be expected, cold-rolled iron sheet loses preferred orientation if heated and cooled through the alpha-gamma transformation temperature.

Some references are appended which will furnish convenient starting points and many additional references for more detailed study.

The Metal Radiography section of this report was prepared by Mr. H. H. Lester and the X-ray Crystallography section was prepared by Mr. L. W. McKeehan and Mr. E. C. Bain.

Respectfully submitted on behalf of the sub-committee,

ZAY JEFFRIES,
Chairman.

APPENDIX

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DISCUSSION

Mr. Malcolm.

MR. V. T. MALCOLM[1] (*presented in written form*).—The report of Committee E-4 on X-ray Metallography, especially the portion covering the applications of metal radiography, which we believe is one of the most important methods of studying foundry practice with regard to special service quality castings, is of much interest. With the recent use of high pressures and elevated temperatures in the steam power plant and oil refinery we have found it of considerable service to study our casting practice by means of the X-ray to prevent failures of castings in service, which failures are likely to result disastrously to both property and life.

Cast-steel valves and fittings designed for high-pressure, elevated-temperature service, whether in oil or steam lines, must conform to rigid specifications and guarantees; and to stand back of these guarantees and assume all that is thereby implied, it is essential that we be in close touch with each step of the manufacturing process, and especially the foundry practice. That steel is steel, seems to be the general idea of quite a few people, but the mere fulfillment of a given set of specifications does not seem to me to be the vital feature.

A recent paper[2] and discussions[3] treated of the use of the X-ray in the testing of steel valves and fittings for the Weymouth Plant of the Boston Edison Co., which is to operate at 1200 lb. pressure and 750° F. temperature. We agree with the several authors[2, 4, 5] of this and other recent papers as to the value of metal radiography. Research work with the X-ray,[4] has already resulted in lengthening the life of certain equipment in use in an oil refinery from 300 to 400 per cent, and the number of fires and accidents resulting from faulty material are believed to have been greatly reduced. The writer has given metal radiography considerable study during the past several years in locating casting faults and improving general casting technique, and in this discussion I should like to recount a few of our experiences. In it all

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[1] Metallurgist, Chapman Valve Manufacturing Co., Indian Orchard, Mass.

[2] Moultrop and Norris, "X-ray Examination of Steel Castings," *Mechanical Engineering*, Vol. 47, Section 2, May, 1925.

[3] V. T. Malcolm and A. Sproat, Discussion on "Methods of Examining Steel Castings by X-rays for Defects," *Mechanical Engineering*, Vol. 47, p. 658, August, 1925.

[4] H. H. Lester, "X-ray Tests as Applied to Problems of the Steel Foundry," *Transactions*, Am. Soc. Steel Treating, Vol. 6, No. 5, 1924.

[5] Keith J. Fanshier, "X-ray Detects Refinery Steel Defects," *The Refiner and Natural Gasoline Manufacturer*, p. 54, July, 1925.

Mr. Malcolm.

we must remember that the method of X-ray testing is a recent development and still in its infancy, and although rapid progress will undoubtedly be made, a word of caution will not be out of place, namely, that the examiner of radiographs should be skilled in foundry technique, and be thoroughly capable of distinguishing between *defects* and *blemishes*.

Defects in steel castings may be listed under the following headings, which in general cover their causes:

1. Piping and other shrinkage cavities due to contraction of steel during solidification;
2. Stresses set up and cracks formed by contraction of steel in solid or semi-solid state;
3. Internal blow holes;
4. Surface blow holes and other external defects.

This listing may be enlarged upon as follows:

1. PIPING AND INTERIOR POROSITY

May be caused by:
- Low-carbon steel;
- Improper pouring temperature;
- Improper and incorrect gating and feeding of castings.

2. CRACKING

May be caused by:
- Improper chemical composition;
- Uneven sections;
- Poor design;
- Sharp corners;
- Uneven cooling after heat treatment;
- Poor welding practice;
- Resistance of cores or molds to metal.

3. BLOW HOLES

May be caused by:
- Gases in metal;
- Imperviousness of sand molds;
- Gas from sand and cores not escaping;
- Oxides in metal caused by improperly made steel;
- Thin slags intimately mixed with molten metal.

4. SURFACE DEFECTS

May be caused by:
- Entrained slag;
- Sand used not being sufficiently refractory causing "burning in" in spots;
- Moist sand causing steam;
- Improper pouring temperature;
- Defective welding.

Mr. Malcolm. It is not our intention to enter into a general technical discussion as to the cause of foundry troubles and their remedies, but a few remarks regarding the investigation and cause of these defects will be in order. Studies of all were made with the X-ray and castings cut up and deeply etched.

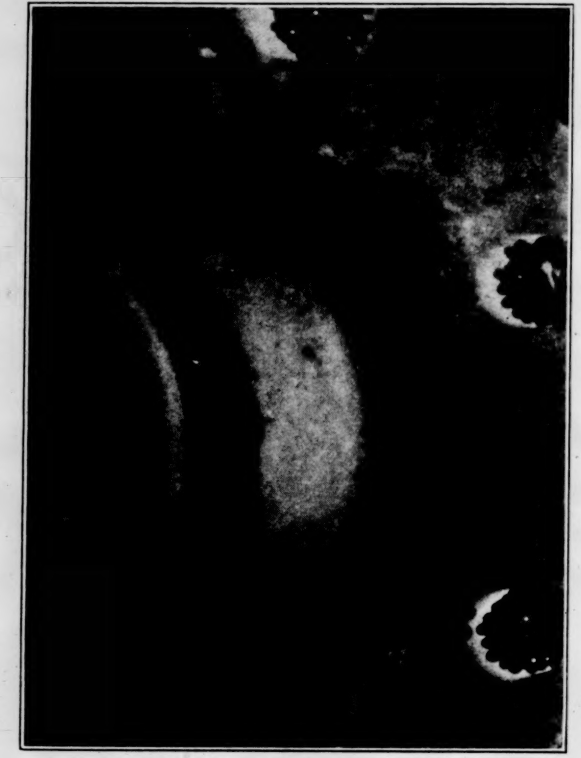

FIG. 1.—Radiograph of Experimental Steel Casting Showing Porosity at Base of Shrink Head.

The several dark ragged holes are the bolt holes in the flange of the casting which were filled with lead while taking the radiograph.

Shrinkage and Porosity.—The explanation of the cause of shrinkage cavities from our studies is in our opinion as follows:

As soon as liquid steel comes in contact with the bottom or sides of the mold, solidification takes place, the steel frozen around the liquid interior consisting of an inner and an outer layer. Steel in all layers tends to contract as it cools, and the outer layer chills much more rapidly than the inner layer, becoming solid almost instantly, with the result that any slag, dirt or loose sand from the

Mr. Malcolm.

sides of the mold becomes entrapped by quick chilling of the steel and causes certain surface defects as well as more deep-seated ones.

As steel freezes, it contracts in volume, and since freezing begins from the outside and progesses inwardly, the crystallization, which begins at the outside, being completed near or somewhat near the center of the mass, due to the temperature gradient of the cooling steel, the contraction takes its effect at the center in the formation of a cavity familiarly known as a pipe. (See Figs. 1 and 2.)

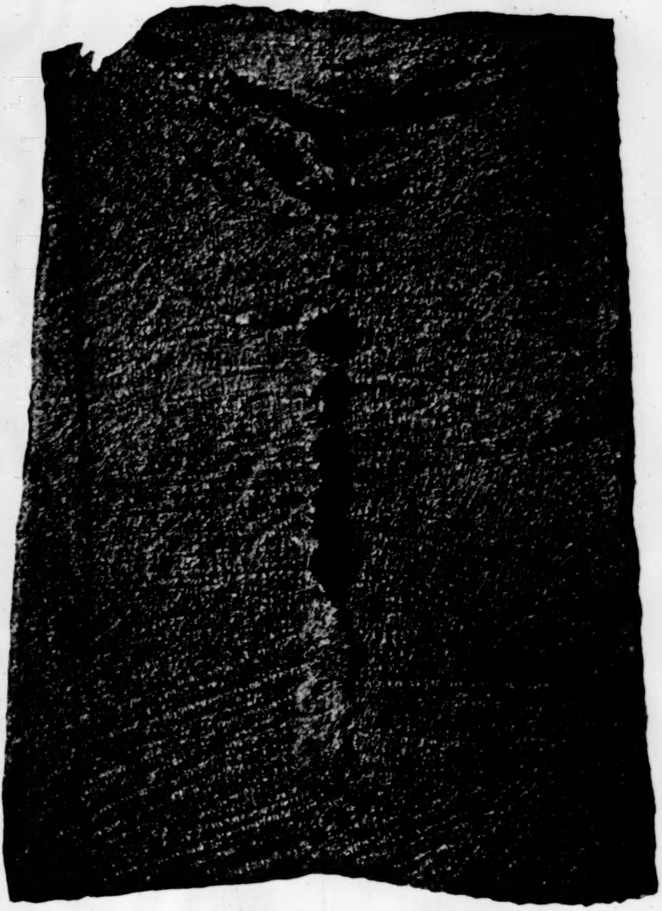

FIG. 2.—Section Cut From Casting and Deeply Etched with 50-per-cent Boiling Hydrochloric Acid.

It will be noted that considerable shrinkage has taken place and that the so-called false "roof" has caved in. Secondary piping and dendritic structure usually accompanying piping are plainly visible. A photomicrograph of a section through this piping shows well-defined dendritic structure. At top it will be noted that the cavity contains some loose sand.

Mr. Malcolm. Piping or shrinkage is bound to occur to a greater or less extent, even in the best of castings. Experienced metallurgists know that the remedy is to control the location and size of these cavities by the proper proportioning and location of gates, heads and runners, for here the laws of fluid pressure are of great importance. Improper application or faulty understanding of the laws of fluid pressure often leads to the adoption of means of feeding castings that result in considerable wastage without any compensating good having been accomplished.

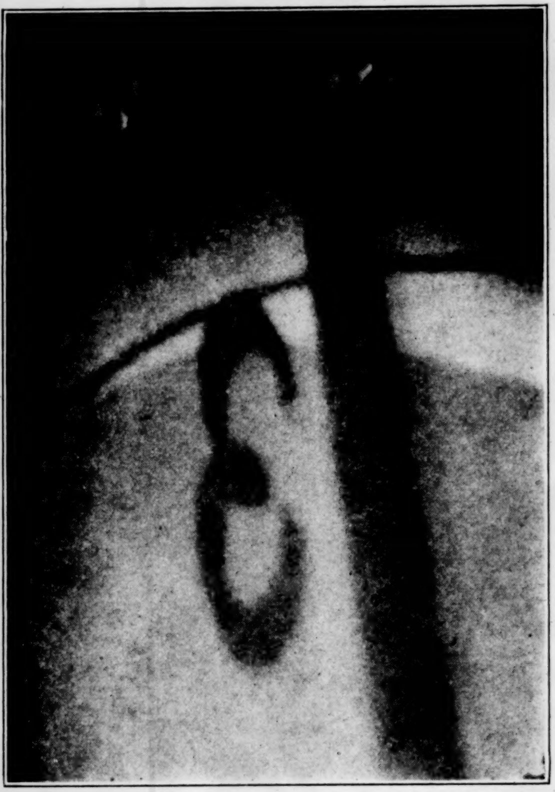

FIG. 3.—Radiograph of Steel Casting Similar to that Shown in Fig. 1.

Here the shrinkage and piping has been eliminated entirely by study of previous results. The location is one at which it is very difficult to eliminate shrinkage as it is the junction between the body section and flange of a steel fitting. Black number and line indicate location of this junction.

Only after careful analysis of the entire situation, by cutting up castings and studying them, may favorable results be obtained. By the use of the X-ray on "pilot" castings, the location and depth of

Mr. Malcolm.

"pipe" may be studied and "heads" moved in such a way as to eliminate piping in stressed parts of the casting. Radiography here is far more effective than cutting castings. The only objection is the cost, as from fifteen to twenty pictures must be made from each casting, which in our case covered about 70 per cent of the total metal surface. Vital sections, such as where the body is joined to flange and bosses, are examined very carefully, for here, if anywhere, defects will take place. (See Fig. 3.)

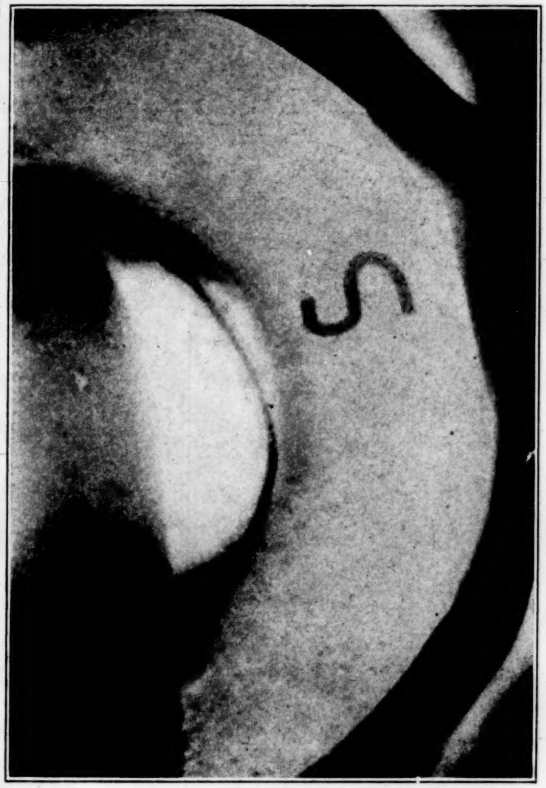

FIG. 4.—Radiograph Showing Capillary Piping.

Freezing starts at some distance from the sink head, and as it progresses, solidification in distant sections takes place and fluid metal must be drawn from nearby sections to make up for this loss, with the result that piping as noted takes place.

We find that steel poured at certain temperatures will "pipe" more below the head than steel poured at other temperatures, and the size and position of this pipe is of great importance in the casting operation. Since piping is caused by shrinkage of fluid metal just

Mr. Malcolm. before or at the moment of solidification, if there is any constriction between the mass of metal in the casting and the "gate," the metal will solidify in the thinner portions before the thicker portion becomes solid. Liquid contraction will go on in the thick part and as no liquid steel is available to fill up the intercrystallin spaces, sponginess or what Lester[1] calls "capillary piping," will result. (See Figs. 4 and 5.) To overcome this condition the metal must cool from the outside to the "head," and behind each crystal as it freezes there must be fluid metal directly connected with the "head" so that there will be no interruption to feeding in of more liquid metal to fill up the spaces caused by liquid contraction.

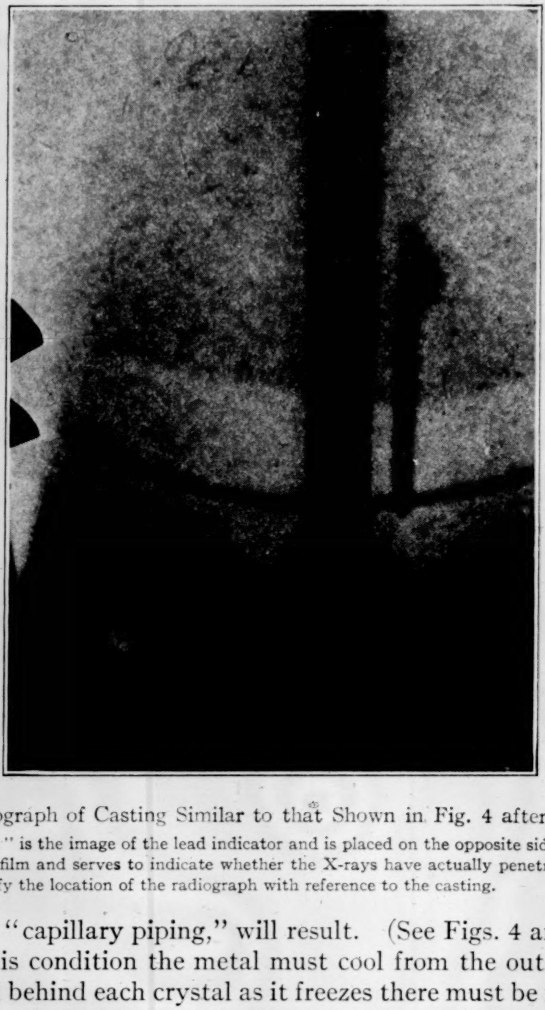

FIG. 5.—Radiograph of Casting Similar to that Shown in Fig. 4 after Remedying.

The figure "1" is the image of the lead indicator and is placed on the opposite side of metal from the photographic film and serves to indicate whether the X-rays have actually penetrated the metal and also to identify the location of the radiograph with reference to the casting.

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[1] H. H. Lester, "X-ray Tests as Applied to Problems of the Steel Foundry, "*Transactions*, Am. Soc. Steel Treating, Vol. 6, No. 5, 1924.

Mr. Malcolm.

In addition to the defects noted, zones of weakness are caused by the interior porosity which is almost always accompanied by a dendritic structure, and, of course, the safety factor is reduced.

Cracks.—Cracks in steel castings are readily understood and present little difficulty if care is taken and the casting is designed correctly, so that thick and thin sections do not join abruptly. Castings sometimes have sharp corners and angles and stresses are set up in the metal due to unequal rates of cooling between thick and thin sections. These stresses sometimes are greater than the strength of the steel and cracks are formed.

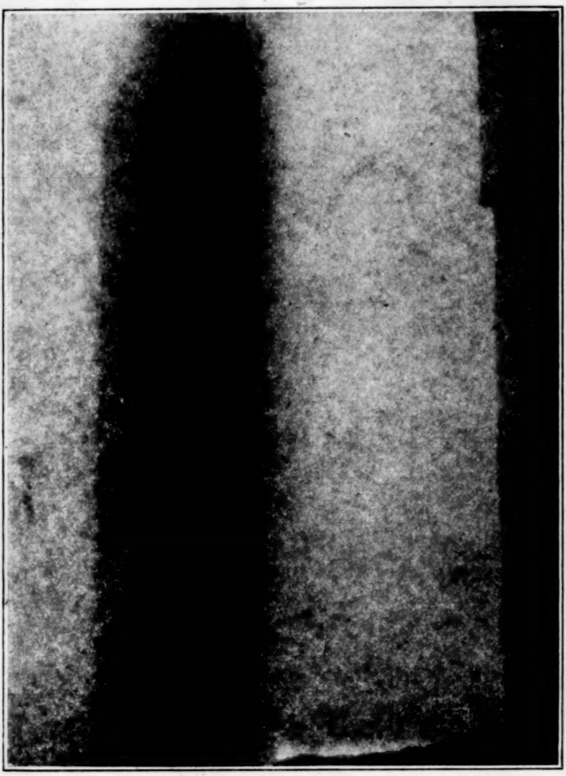

FIG. 6.—Radiograph Showing a Crack in a Steel Casting Not Visible on the Surface.

Cracks are due to other causes: namely,

To improper chemical composition, such as high sulfur, oxides of iron and manganese which effect concentration of impurities along the grain boundaries, causing the steel to crack easily;

Mr. Malcolm. Rapid cooling after heat treatment causes cracking;

Hard cores may set up stresses in castings and cause cracks;

Faulty welding may cause hair line cracks, not visible to the naked eye.

All types of cracks are readily determined by means of radiography. (See Figs. 6, 7, 8 and 9.)

Metal Defects.—Rarely is metal the cause of interior porosity or defects, but some time ago, the writer claimed that metal, if improperly made, could be the source of defects, perhaps bad ones. In an endeavor to prove this, we purposely made a small heat of effervescent steel, or steel which had been improperly deoxidized, with the results shown in Fig. 10.

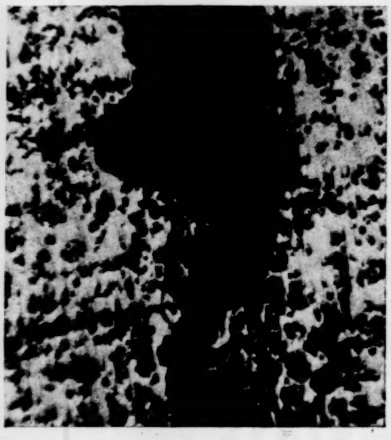

FIG. 7.—Macrograph of Section Cut From Casting Shown in Fig. 6 and Deep-Etched with 50-per-cent Boiling Hydrochloric Acid. (× 10.)

Large crack and porous sections adjacent to crack are well defined.

After careful study we reached the conclusions that effervescence in steel may be defined as an escape from the molten metal of gases, mostly carbon monoxide, in myriads of bubbles, their escape from the metal tending to keep it in violent motion with a churning effect as the metal freezes from sides to center. These bubbles throw off brilliant sparks from the liquid steel, becoming more infrequent as the steel freezes. Effervescence in steel will cause a "kick back" in the sink head and gives rise to the so-called "cauliflower head." The blow-hole cavities, which may be seen in the radiograph, are due to the presence of gas, the oblong ones are caused by improperly deoxidized metal and the spherical ones by steam generated from a damp

Mr. Malcolm.

mold at the time of casting. The oblong holes are caused by a chemical reaction taking place in the metal due to its incomplete deoxidation, and are silvery white and contain a black deposit which upon chemical analysis is found to be iron oxide, together with some silica; while the metal adjacent contained considerable so-called "dirt" which in reality was oxide. The bubbles of the oxides are very minute and bubbles of carbon monoxide form around them, which, failing to rise through the molten metal, cause sponginess in the casting. The explanation, we believe, is, that when "wild" steel containing iron and manganese oxides is poured into a sand mold, a reaction takes place between these oxides and the carbon in the steel while the steel is in a semi-solidified condition. This results in the formation of carbon-monoxide gas, which is forced outward through the pasty steel by expansion, but which cannot escape to the "sink head" because the neck has already been chilled, and as further feeding of liquid steel is

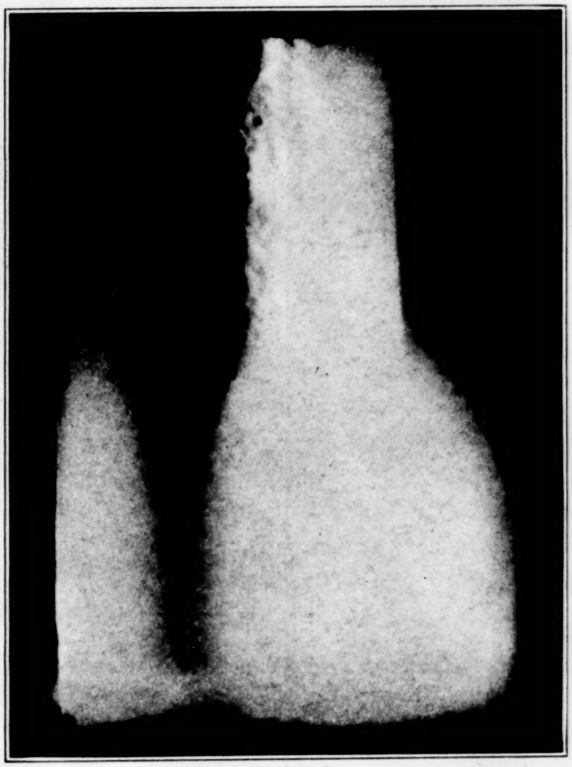

Fig. 8.—Radiograph Showing the Location of a Faulty Weld in a Steel Casting.

Mr. Malcolm. not possible, the gas remains entrapped in the casting, mostly below the "sink heads."

All metal, no matter how thoroughly deoxidized it may be, contains gases in solution (converter steel the most, electric steel very little), and other gases are generated from the molding material, such as the sand in the molds and cores. These gases must escape through the metal and through the sand in order to obtain good castings.

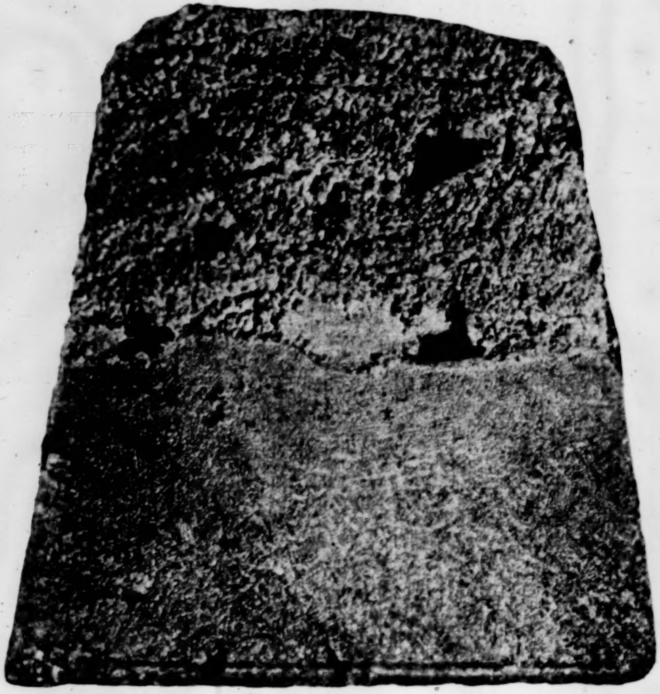

FIG. 9.—Macrograph of Section of Cut Through Casting Shown in Fig. 8. Etched with 50-per-cent Boiling Hydrochloric Acid.

It may be seen that the metal was burned before it was melted in the welded section and that the weld did not join the original section but simply formed a new layer.

Lester[1] described in his valuable paper the bad effect of gases, dirt and sand in metal and our work has taught us the value of radiography in locating these troubles and has pointed the way to the solution of these problems.

Dirt, Sand, Etc.—One of the most valuable uses of radiography is the determination of the amount of sand entrapped in castings and of

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[1] H. H. Lester, "X-ray Tests as Applied to Problems of the Steel Foundry," *Transactions*, Am. Soc. Steel Treating, Vol. 6, No. 5, 1924.

Mr. Malcolm.

the amount of gas contained therein, due to the imperviousness of molds, and moist and weak sands. As a result of our study of castings by radiography, a complete sand control laboratory was established for routine control of all molding sands.

By such routine control work on sand, we are enabled:

1. To determine the amount of moisture in sand and to make necessary correction before the sand is made into molds.

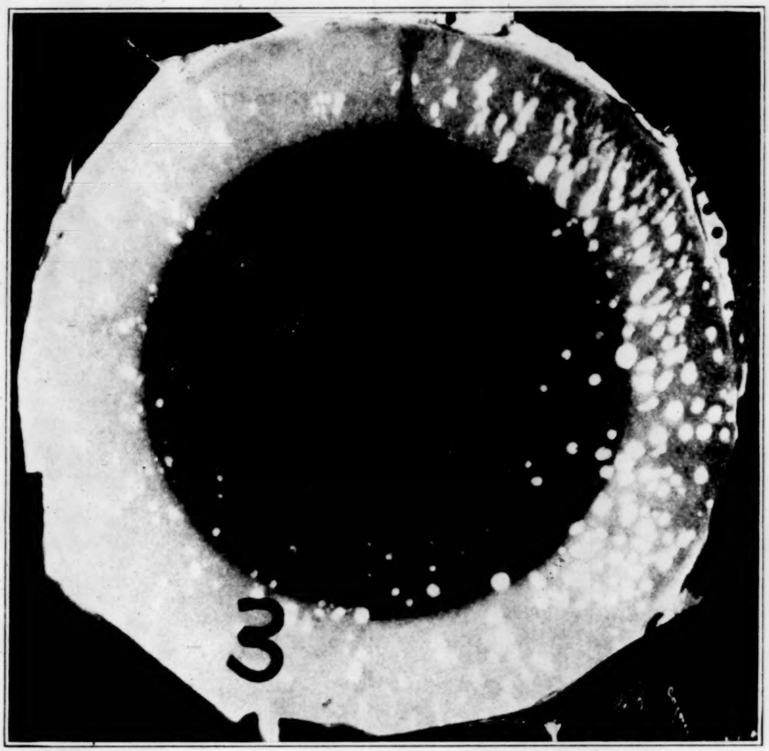

FIG. 10.—Radiograph of Experimental Steel Casting Showing the Effect of Improperly Deoxidized Metal.

2. To determine the actual resistance of sand to the passage of gases, which enables us to establish a standard permeability for sand.
3. To determine the actual strength of the sand so there will be no breakage of molds during casting.
4. To determine the refractoriness and durability of the sand, so as to enable it to withstand the hot metal.

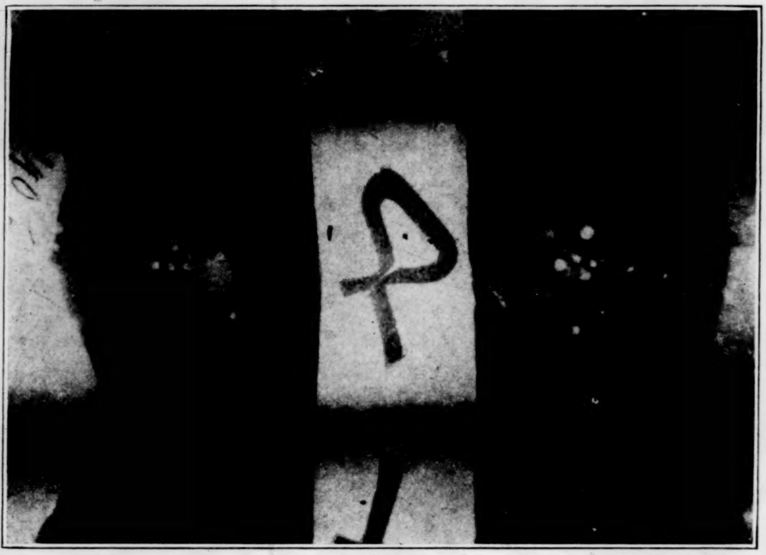

FIG. 11.—Radiograph of Experimental Steel Casting Made in "Green Sand" and Showing Blow Holes Due to Steam Being Injected Into the Metal From a Damp Mold.

The sand in this case was purposely dampened to a considerable extent to study the effect of moisture in sand.

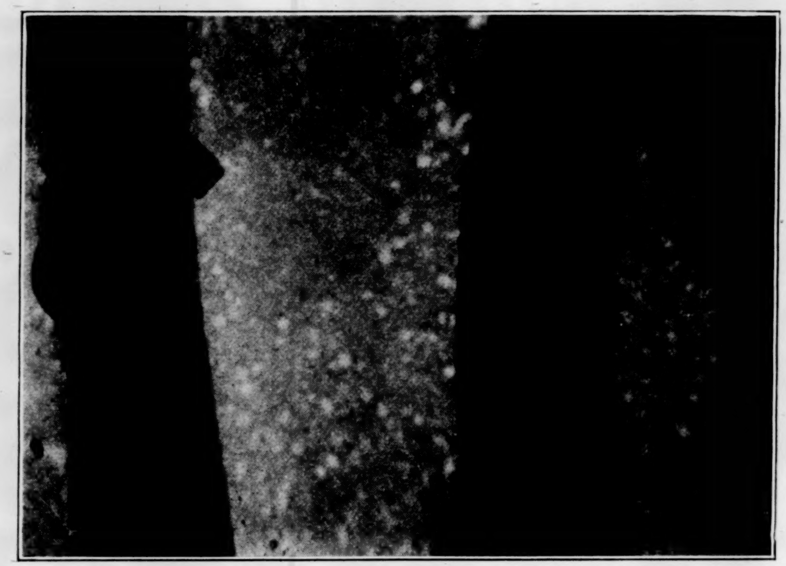

FIG. 12.—Radiograph of Experimental Steel Casting Showing Gas Holes that are Entrapped in the Metal and Entirely Due to the Close Packing and Hard Ramming of the Mold.

The sand used in this case was purposely rammed hard in order to study the effect of permeability of sand.

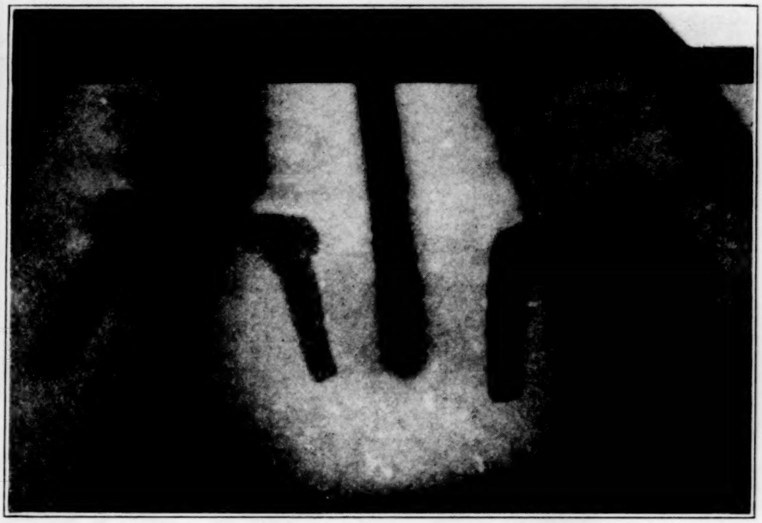

FIG. 13.—Radiograph Showing Effect of Washing Sand From the Mold and Runner During the Casting Operation, the Sand Becoming Entrapped in the Molten Metal.

The white spots are sand, which is shown by photomicrographs to have fused into the metal.

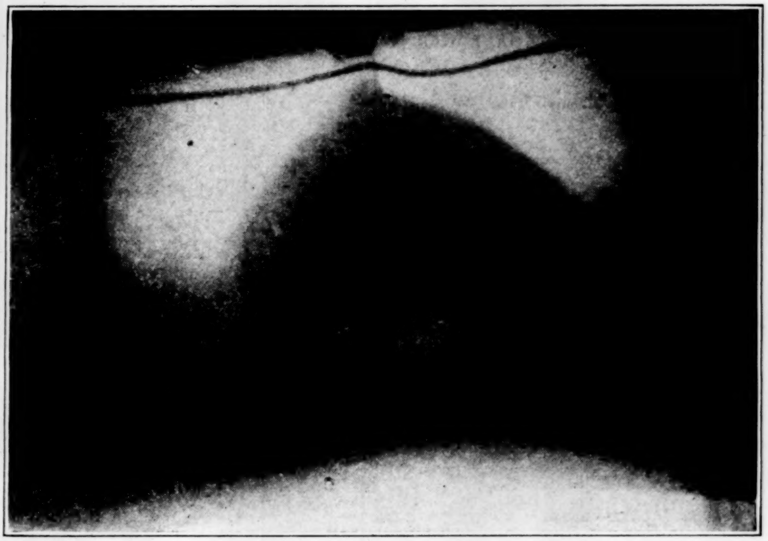

FIG. 14.—Radiograph of Casting Similar to Those Shown in Figs. 11, 12 and 13, but With the Sand Conditioned Correctly.

Mr. Malcolm Certain standards set up for this work, which have proved satisfactory, are strictly adhered to and casting losses reduced to a minimum. (See Figs. 11, 12, 13 and 14.)

Conclusions.—In summing up the cause of foundry defects we would say that such defects as entrapped gas or blow holes, sand, slag and dirt inclusions may be eliminated entirely by close study and careful attention to foundry practice and that "pipe cracks" and cavities can be controlled by proper design and knowledge of castings at hand.

We have great faith in what radiography has done and will do to insure the integrity of castings for high-pressure, elevated-temperature service. We do not recommend the X-ray as a routine test, but do believe in its value as a method of investigation.

Mr. St. John. MR. ANCEL ST. JOHN.[1]—I want to comment on the radiographic portion of the report and upon some points that Mr. Malcolm has just made, namely, that it is practically impossible to make a steel casting without having some sort of cavities in it, and that the question as to whether or not castings are such as may be used in service, is a question of whether the cavities which are bound to exist are kept within certain tolerance limits as regards size and location. The radiographic examination, using the photographic method, certainly discloses all the information that it is possible to obtain with the X-ray concerning these factors. But I am persuaded that with the more powerful tubes at present coming on the market, that is, more powerful with respect to the quantity of X-rays of the given penetrating power that are produced by the tubes, we will find that a great many of the defects, and, perhaps, all of the defects which are so great as to render necessary the rejection of the casting, can be disclosed on a fluorescent screen and observed at once, without going through the procedure of photographic examination. That in itself will not only speed up X-ray inspection, but will reduce the cost very considerably, because in radiographic examination, using the photographic method, you are confronted with an irreducible minimum of expense of at least 50 cents per square foot for photographic material, and labor on top of that. In order to find out whether my idea in regard to this is correct, I have already instituted what I hope is going to be an extensive investigation of that very point; carrying it out by means of securing from the manufacturers of steel castings certain representative poor or rejected castings, examining these with the photographic method and then examining them with the fluorescent screen. If they are castings which can be subjected to hydrostatic tests, we will submit them to such tests both before and after the examination, in order to correlate the X-ray findings with the findings as given by other methods of test. I believe we will find with respect to steel and other metal castings that there are a great many types of castings, at least, where the examination by the fluorescent screen of pilot castings in the development, or of every tenth casting in the routine procedure, or even carrying on to the point in certain cases where every single casting for important work is examined individually, will be made a perfectly practical proposition. **Mr. St. John.**

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[1] Consulting Physicist, New York City.

MR. T. MCLEAN JASPER[1] (*by letter*).—I should like to add to the report on the use of X-rays for investigation of metals, a small amount of experience obtained at the University of Illinois on the problem of detecting the beginning of fracture in steel under fatigue. This work was done in the Physics Laboratory by Mr. Knipp in conjunction with the test party of the Fatigue of Metals Investigation in charge of Mr. H. F. Moore. **Mr. Jasper.**

It was found that small fine hair cracks which were discernible by the use of the eye or a small hand glass did not show up in the X-ray picture as it was hoped they would. This suggests that there is a minimum crevasse or cavity width, if it might be so called, which this X-ray method will show up. It is suggested that this minimum cavity width will depend on the thickness and on the density of the material under investigation.

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[1] Engineer of Tests, Fatigue of Metals Investigation, University of Illinois.

REPORT OF COMMITTEE E-6

ON

PAPERS AND PUBLICATIONS

During the year the Committee on Papers and Publications has held one meeting, in February, 1925, at which the general arrangements for the program for the annual meeting were discussed and all offers of papers passed upon for acceptance in accordance with the rules. Since that meeting the Advisory Committee has acted for the committee on all matters relating to the program.

Probably the outstanding feature of the work of the committee was the necessity of greatly curtailing the volume of committee reports and papers to be published this year. This was made necessary by the unprecedentedly large volume of the 1924 Proceedings, and the committee was under instructions from the Executive Committee to keep the volume of Proceedings within such limitations as would meet the 1925 Budget. The committee established the policy of accepting the maximum number of papers and asking the authors to condense them as much as possible, so that the whole field of activities of the Society might be fairly well covered. In this the committee received the hearty cooperation of the various authors so that the number of pages of the technical papers accepted for publication is well within the estimate made by the committee.

It is evident that the Society's business as represented by the committee reports, especially new and revised standards, must be provided for in the publication program. However, with the cooperation of the committee officers it has been found possible to condense certain committee reports, as for example by the omission of progress reports that can be held over for later publication.

There are scheduled on the program 35 committee reports and 26 papers. Thirty-three reports and 20 papers have been preprinted, comprising, respectively, 545 and 216 pages, or a total of 761 pages. It is estimated that the material accepted for publication but not preprinted will total 90 pages.

The committee has given careful consideration to the details of presentation of reports and papers, and particularly the time that could be allowed for their presentation at the meeting. Several suggestions respecting the reading of papers, use of lantern slides, and the like, have been made to the authors of papers. In addition, a time schedule for each session has been carefully worked out and has been placed by the president in the hands of each presiding officer. Each committee chairman and author of a paper has been advised of the time allotted for the presentation of his report or paper and has been asked to keep within that time. Of course, irrespective of any time schedule, time must be taken actually to conduct the Society's business of receiving and acting upon committee reports.

Although the program for this annual meeting is not so large as that for last year, it will be found to contain many matters of interest and importance both among the work of our committees and among the technical papers presented by the members.

The plan of parallel sessions with afternoons free for committee meetings and recreation, inaugurated so successfully last year, has been continued. With the fewer number of sessions, however, it has been arranged that the opening and closing sessions of the Society are not parallel sessions as was necessary last year, the committee feeling that it is very desirable that at these sessions the members should be assembled in one session and not divided between two.

It is appropriate to mention several features introduced into the Society's regular publications for 1924. For the first time, a complete list of the standards and tentative standards was published in the Proceedings so that those who use that volume will now find references to all of the Society's standards and tentative standards. With the Book of Standards was included a suitable foreword explaining the plan of publishing A.S.T.M. standards and also a list of the tentative standards of the Society in pamphlet form. After each annual meeting the list of tentative standards is changed and a revised pamphlet will then be included with copies of the Book of Standards.

For the first time, the Book of A.S.T.M. Tentative Standards was completely indexed. A list of the standards of the Society has been included in this volume for some time.

Respectfully submitted on behalf of the committee,

C. L. WARWICK,
Chairman.

REPORT OF COMMITTEE E-8
ON
NOMENCLATURE AND DEFINITIONS

The duties of Committee E-8 during the past year have consisted for the most part in exercising an editorial review of definitions presented before it and in the correlation of definitions of interest to more than one standing committee. The definitions involved are both those appearing in the standards and tentative standards of the Society and those in a formative stage referred to Committee E-8 for review by the standing committee interested. The correlation of definitions of interest to more than one standing committee has been handled by sub-committees consisting of the representatives of the interested committees. The following is the list of the present sub-committees of Committee E-8, organized for this purpose:

On Specific Gravity (F. M. Farmer, Chairman).
On Definitions of Terms Relating to Cementitious Materials (L. I. Neale, Chairman).
On Definitions of the Terms Sand and Aggregate (L. R. Ferguson, Chairman).
On Definitions of the Terms Sieve and Screen (L. R. Ferguson, Chairman).
On Definitions of Loam, Silt, Gravel and Related Terms (L. R. Ferguson, Chairman).
On Definitions of Moisture, Ash, Volatile Matter and Fixed Carbon (H. C. Porter, Chairman).

One sub-committee, the Sub-Committee on Definition of Clay, under the chairmanship of A. S. Watts, has been discharged, since this sub-committee had completed the work assigned to it. This sub-committee had prepared definitions for the terms "clay," "surface clay," "fire clay," "shale" and "clay separate" which were adopted at the last annual meeting of the Society.

The personnel of the Sub-Committee on Definitions of Moisture, Ash, Volatile Matter and Fixed Carbon has been enlarged by the addition of representatives of Committees D-7 on Timber and D-13 on Textile Materials. The organization of a new Sub-Committee on the Definition of the Term Concrete has been authorized, but organization has not yet been effected. Mr. L. R. Ferguson has accepted the chairmanship of this sub-committee.

The work of Committee E-8 as well as that of its sub-committees is handled so far as possible by correspondence. Following the precedent of the past two years only one meeting of the general committee has been held during the year, this meeting having been held on April 30, 1925, to act upon the approval of definitions going to standard and to take action upon the recommendations of the several sub-committees. The Advisory Committee has held two meetings, in November, 1924, and in April, 1925.

The attentions of Committee E-8 are of necessity largely confined to those definitions having a tentative or sub-tentative (formative) status. Some progress, however, has been made in the review of definitions contained in present standards of the Society, which were adopted before Committee E-8 began its duties as a receiving body. The standard definitions of terms relating to paint specifications were given some review prior to the 1924 annual meeting, since a number of definitions approved by Committee E-8 in 1922 were being combined with those adopted in 1915 before the organization of Committee E-8. The definitions of terms relating to timber are receiving a comprehensive review on the part of Committee D-7 in cooperation with the Central Committee on Lumber Standards. The recommendations of Committee D-7 will result in the replacement of many of the present standard definitions of terms relating to timber. In addition, a number of standard definitions have been revised through the regular Society procedure of having tentative standards published and finally adopted to replace the standards. Among these might be mentioned the definitions of terms relating to magnetic testing, the definitions of terms relating to clay, and the definitions of terms relating to the verification of testing machines. A definition for the term "screen" is now being presented, which, if adopted, will supersede two standard definitions, namely, the definitions of the terms "sieve" and "screen" appearing in the Standard Definitions of Terms Relating to Materials for Roads and Pavements (D 8).

There still remain, however, a considerable number, approximately 275, of standard definitions that have received no detail consideration on the part of Committee E-8. The committee hopes to report progress on this work at the next annual meeting.

DEFINITIONS OF TERMS APPROVED BY COMMITTEE E-8

In 1922, Committee E-8 approved nine definitions of terms relating to paint and the definition of the term "A.S.T.M. Paraffin Wax

Melting Point." In 1923, four definitions relating to rubber analysis were approved. In 1924, a number of terms were approved, seven definitions of terms relating to magnetic testing, twenty-three definitions of terms relating to hollow tile, three definitions of terms relating to gypsum, six definitions of terms relating to verification of testing machines, eleven definitions of terms relating to metallography, the definitions for the terms "clay," "clay separate" (the definitions for "surface clay," "fire clay" and "shale" are included among those relating to hollow tile), and the definition of "A.S.T.M. Petrolatum Melting Point." The complete list of the 67 terms approved by Committee E-8 is as follows:

Aging Coefficient
Alloy
Alpha Brass
A.S.T.M. Paraffin Wax Melting Point
A.S.T.M. Petrolatum Melting Point
Beta Brass
Book Tile
Calcined Gypsum
Cells
Clay
Clay Separate
Coercive Force
Core Loss
Correction
Crushed Gypsum
Drying Oil
Enamel
End-Construction Tile
Equi-axed Grain
Error
Etching Reagent
Filler
Fire Clay
Foundation Tile
Furring Tile
Grain
Grain Size
Gypsum
Hollow Floor Tile
Hollow Tile
Hollow Tile Fireproofing
Lake
Length
Load Bearing Wall Tile
Loading Range
Macrograph
Magnetic Permeability
Magnification
Metal
Micrograph
Non-Drying Oil
Normal Induction
Organic Acetone Extract
Partition Tile
Percentage of Error
Porous Hollow Tile
Residual Induction
Salt-Glazed Tile
Saponifiable Acetone Extract
Scoring
Semi-Drying Oil
Shale
Shell
Side-Construction Tile
Size
Split Tile
Standard Core Loss
Surface Clay
Testing Machine
Thickness
Tolerence
Toner
Total Waxy Hydrocarbons
Unsaponifiable Resins
Varnish
Webs
Width

A number of definitions have been withdrawn. In other instances the term defined has been changed somewhat but the definition has been retained in substantially the same form. In some cases the definition has been withdrawn as such, but the essential feature of the definition has been resubmitted as a paragraph on scope in specifications, or as notes to other definitions. In all 145 tentative definitions of those submitted prior to 1924 still remain before Committee E-8 for consideration. At the 1924 annual meeting, 5 additional terms were submitted as tentative. The total number of tentative definitions of terms before Committee E-8 at the present time is accordingly 150.

Tentative Definitions of Terms Submitted Prior to 1924

Of the 145 terms remaining before Committee E-8 after the 1924 annual meeting some few are now being recommended for advancement to standard. Still others are remaining as tentative but have been brought into agreement with the suggestions of Committee E-8. Still others are still in the hands of appropriate sub-committees and will require additional work before satisfactory definitions can be reported. The action taken to date on each of the terms is as follows:

Terms Relating to Coke (D-121).—The 15 terms defined by Committee D-5 on Coal and Coke are as follows:

- Coke
- Beehive Coke
- By-Product Coke
- Gas House Coke
- Coke Breeze
- Domestic Coke
- Foundry Coke from Beehive Ovens
- Foundry Coke from By-Product Ovens
- Furnace Coke from Beehive Ovens
- Furnace Coke from By-Product Ovens
- Dry Coke
- Moisture
- Ash
- Volatile Matter
- Fixed Carbon

The definitions of the terms "moisture," "ash," "volatile matter" and "fixed carbon," with certain revisions suggested by the Editorial Committee, are still before a representative sub-committee of Committee E-8 under the chairmanship of H. C. Porter. The definitions of the remaining 11 terms were reviewed by the Editorial Committee and certain rewordings suggested to Committee D-5. Committee D-5 submitted revised definitions for these terms at the 1924 annual meeting, bringing the definitions in closer agreement with the suggestions of the Editorial Committee.

Terms Relating to Coal (D 142).—The 6 terms presented by Committee D-5 on Coal and Coke are as follows:

- Moisture
- Ash
- Volatile Matter
- Fixed Carbon
- Proximate Analysis
- Ultimate Analysis

The terms "moisture," "ash," "volatile matter" and "fixed carbon," as in the case of these terms as defined as relating to coke, are under consideration by a special sub-committee under the chairmanship of H. C. Porter. A revision of the definition for "proximate analysis" was presented in 1924 by Committee D-5 and a revision of the term "ultimate analysis" is being recommended by Committee D-5 this year.

Terms Relating to Gypsum (C 11, C 22, C 23, C 36, C 37, C 52).—The 12 terms defined by Committee C-11 on Gypsum as submitted in 1921 in the Tentative Definitions of Terms Relating to the Gypsum Industry (C 11) are as follows:

Accelerator
Aggregate
Binder
Cement
Consistency
Keene's Cement
Mortar
Plaster
Plasticity
Retarder
Stucco
Wood Fiber

The following definitions appear in the specifications prepared by the committee:

Gypsum
Calcined Gypsum
Gypsum Wall Board
Gypsum Plaster Board

The term "Keene's Cement" was withdrawn by Committee C-11 at the suggestion of Committee E-8 in 1922. A definition for the term "lath" was added to the definitions by Committee C-11 in 1922 and a definition for "gypsum partition tile or block" was added in 1923. The definitions for the terms "gypsum," "calcined gypsum," "crushed gypsum" (submitted in 1924), "gypsum wall board," and "gypsum plaster board" were approved by Committee E-8 in 1924. The definitions for the terms "gypsum wall board" and "gypsum plaster board," however, were not accepted at the 1924 annual meeting and were eliminated from the Tentative Definitions of Terms Relating to the Gypsum Industry (C 11). They were retained, however, as a matter of expediency, in Specifications C 36 and C 37, respectively, with the understanding that they would receive a critical review by Committee C-11 during the year.

The definitions of the terms "consistency" and "plasticity" are receiving the consideration of a Sub-Committee, of Committee E-1, on Methods of Testing. This sub-committee is presenting as information a definition for the term "consistency" which appears in the 1925 report of Committee E-1. The term "aggregate" has been referred for development to the Sub-Committee on Sand and Aggregate under the chairmanship of L. R. Ferguson, which sub-committee is this year presenting a definition for the term "aggregate" for publication as information. The remaining terms are now before the Sub-Committee on Definitions of Terms Relating to Cementitious Materials under the chairmanship of L. I. Neale, as follows:

Accelerator
Binder
Cement
Lath
Mortar
Plaster
Retarder
Stucco
Wood Fiber

Terms Relating to Lime (C 51).—The 5 terms defined by Committee C-7 on Lime presented in 1922 are as follows:

Quicklime
Hydrated Lime
Lump Lime
Lump Lime Screened
Pulverized Lime

Committee E-8 gave these definitions an editorial review and since Committee C-7 appeared to be the only standing committee interested, the definitions were referred to that committee with a number of suggestions for rewording. The definitions were revised and resubmitted in 1924 by Committee C-7 in substantially the form recommended by Committee E-8. The definitions, however, are not being recommended for advancement to standard this year and accordingly no letter ballot has been conducted in Committee E-8.

Terms Relating to Waterproofing Materials (D 147).—The three terms submitted by Committee D-8 on Waterproofing Materials are as follows:

Bituminous Grouts
Bituminous Mastics
Mastic Cake

The terms being defined after receiving further consideration in Committee D-8 were changed to "bituminous grout," "asphalt mastic" and "asphalt mastic cake," respectively. Committee E-8 has been in correspondence with Committee D-8 suggesting a number of changes in wording and receiving a number of suggestions from Committee D-8. Substantial agreement has now been reached on these definitions. The revised wordings are being presented this year. Since, however, Committee D-8 is not recommending the advancement to standard of these definitions they have not been referred to the membership of Committee E-8 for approval.

Definitions of Terms Relating to Textiles.—A number of terms relating to textiles were submitted in 1921 and additional terms were submitted in 1922 and 1923. The complete list of terms is as follows:

Those appearing in the Tentative Definitions of Terms D 123 are as follows:

Alberts
Army Duck
Balata Belt Duck
Clay (a kind of weave)
Crimp
Drills
Enameling Duck
Hose Duck
Jeans
Numbered Duck
Off-Square
Oil Belt Duck
Rubber Belt Duck
Serge
Single Filled or Flat Duck
Tire Builder Fabric
Tire Cord Fabric
Twill

Those appearing in Specifications D 122 are as follows:

MAJOR DEFECTS	MINOR DEFECTS	SPECIAL DEFECTS
Thick Places	Thick Places	Oil Spot
Thin Places	Thin Places	Mixed Filling, Inferior Grade
Missing Warp Thread	Slack Threads	Smashes
Pucker	Tight Threads	Imperfect Selvage
Bad Start-up	Slug	Wrong Ply Filling Threads
	Sewed Thread	
	Knot	

MINOR DEFECTS

Mispick or Broken Pick
Float
Reed Mark
Split or Chafed Yarn
Dirty Yarn
Wrong Ply Warp Thread
Wrong Draw

Those appearing in Specifications D 179 are as follows:

MAJOR DEFECTS

Thick Places
Thin Places
Missing Cord
Knots of Finished Cord

MINOR DEFECTS

Thick Places
Thin Places
Slack Cord
Tight Cord
Slug
Mispick or Broken Pick
Float
Chafed Cord
Dirty Yarn
Wrong Ply Cord
Wrong Draw
Corkscrew Twist
Kinks

SPECIAL DEFECTS

Oil Spot
Smashes
Imperfect Selvage
Baggy Fabrics
Irregular Picks
Mixed Cords
Defective Splice

In addition a definition for the term "direction of twist" appears in Specifications D 180.

The definitions appearing in Specifications D 122 and D 179 are of the same general type; in fact, in some instances the same definition appears in both standards. They have accordingly been considered conjointly. The definitions are quite special and of interest almost exclusively to Committee D-13. After receiving editorial review, they were discussed with that committee. They are now being recommended for advancement to standard by Committee D-13. They have been referred to letter ballot in Committee E-8, the results of this letter ballot being as follows: Advisory Committee, 3 affirmative, 1 negative, 3 not voting, total 7. Standing Committees, 12 affirmative, 0 negative, 26 not voting, total 38.

The definition for "direction of twist" has been revised by Committee D-13 in the light of comments submitted by Committee E-8. A definition for the same term with the identical wording appears in Specifications D 203. A definition for "direction of twist" but with a meaning directly opposite to that appearing in Specifications D 180 and D 203 appears in Specifications D 204. The Specifications D 203 and D 204 were presented in 1924.

The specifications in which the definitions for "direction of twist" appear are being recommended by Committee D-13 for advancement to standard. Committee E-8 does not look with favor upon the acceptance of two definitions diametrically opposite in meaning for the same term. However, it does not wish to interpose an objection to the advancement to standard of the tentative specifications in question. It accordingly has approved the form of the revised wording for the "direction of twist" and recommends the addition of a note calling attention to the discrepancy existing in the two wordings of the definition. The definition has accordingly been included with the other definitions relating to textile materials mentioned above as having been referred to letter ballot in Committee E-8.

Committee D-13 is recommending the continuance as tentative of the Tentative Definitions of Terms Relating to Textile Materials (D 123) since it has under contemplation a general rearrangement of all definitions of terms relating to textile materials in the form of a glossary. While the definitions are generally satisfactory, they have not been referred to the membership of Committee E-8 in view of their continuance as tentative.

Terms Relating to Paint.—Committee D-1 on Preservative Coatings for Structural Materials presented in 1923 definitions for the terms "toughness" and "elasticity of paint and varnish films" to be added, when adopted, to the Standard Definitions of Terms Relating to Paint Specifications (D 16). They have been reviewed by Committee E-8 and are being revised this year by Committee D-1 in the light of the suggestions received from Committee E-8. The term "elasticity of paint and varnish films" has been changed to "elasticity" and is being defined in regard to its relation to paint and varnish films.

Miscellaneous Terms.—There are 5 terms classed as miscellaneous:

Ductility (D 113)
A.S.T.M. Lubricating Oil Precipitation Number (D 91)
Cloud Point (D 97)
Pour Point (D 97)
Specific Gravity (D 70, D 71)

The definition of "ductility" (as given by Committee D-4 on Road and Paving Materials) as applying to the ductility of asphalt cement was referred back to Committee D-4, since it was felt by Committee E-8 that the definition as worded could scarcely be considered a definition of ductility but rather a description of how that property is determined. Committee D-4 is contemplating revising the methods in which this definition appears. The definition of "ductility" will come up for consideration in conjunction with this general revision. Definitions for terms relating to specific gravity are being submitted for publication as tentative by the Sub-Committee, of Committee E-8, on Specific Gravity, under the chairmanship of F. M. Farmer, as mentioned below in connection with the work of that sub-committee. Definitions for the terms "cloud point," "pour point" are still under consideration by the appropriate sub-committees of Committee D-2. The term "A.S.T.M. Precipitation Number of Lubricating Oils" has been approved by Committee D-2 and is still under consideration by Committee E-8.

TENTATIVE DEFINITIONS OF TERMS PRESENTED IN 1924

Only a few new definitions were presented in 1924 as follows:

Terms Relating to Lime.—A definition for the term "in bulk" was added by Committee C-7 to the Tentative Definitions of Terms Relating to Lime (C 51). This definition has been reviewed by Committee E-8, as a result of which Committee E-8 questions the utility of and need for this particular definition and has referred the matter to Committee C-7 for advice.

Terms Relating to Gypsum.—Committee C-11 submitted a new definition for "crushed gypsum" for addition to the terms appearing in the Tentative Definitions of Terms Relating to the Gypsum Industry (C 11). This definition was reviewed by Committee E-8 and the definition approved in 1924.

Terms Relating to Textiles.—A definition for the term "direction of twist" was presented by Committee D-13 in its Specifications D 203 and D 204. This definition, after suitable revision, is being recommended for advancement to standard with the approval of Committee E-8 as mentioned above under terms relating to textiles.

Definition of the Term Sand.—A definition for the term "sand" was presented in 1924 by Committee E-8. The recommendations concerning this definition are recorded below where the activities of the Sub-Committee on Sand and Aggregate are reported.

Terms Relating to Methods of Testing.—Committee E-1 submitted in 1924 a definition for the term "compressive strength" in its Tentative Methods of Compression Testing of Metallic Materials (E 9). This definition has received the review of Committee E-8 and a revision is being recommended by Committee E-1 this year.

TERMS WITHDRAWN

A number of definitions have been withdrawn from time to time by the standing committees responsible for them. Other terms have been discontinued and in some instances a more appropriate term has been defined than the one previously employed. In order to make a matter of record of the various terms that have come up for consideration in Committee E-8 the following list is given of the discontinued terms and definitions.

Achromatic.—Withdrawn by Committee E-4.

Achromatic Objectives.—Withdrawn by Committee E-4.

Apochromatic.—Withdrawn by Committee E-4.

Back-up Tile.—Withdrawn by Committee C-10.

Bituminous Mastics.—Withdrawn by Committee D-8 and resubmitted as a definition of "asphalt mastic."

Boiling Point.—(D 2) Classified by Committee E-8 as not a definition.

Bulk.—Withdrawn by Committee D-1.

Clay.—Withdrawn by Committee C-4 and resubmitted as a definition for "surface clay."

Creosote Oil.—(D 8) Classified by Committee E-8 as not a definition.

Distillation Loss.—(D 2) Classified by Committee E-8 as not a definition.

End Point.—(D 2) Classified by Committee E-8 as not a definition.

Equi-axed.—Withdrawn by Committee E-4.

Fire Point.—(D 2) Classified by Committee E-8 as not a definition.

Flash Point.—(D 2) Classified by Committee E-8 as not a definition.

Floor Tile.—Withdrawn by Committee C-10 and resubmitted as a definition for "hollow floor tile."

Heavy Strip.—Withdrawn by Committee D-13.

Illuminator.—Withdrawn by Committee E-4.

Intercept Method for Grain Count.—Withdrawn by Committee E-4 and resubmitted as a definition for "grain size."

Keene's Cement.—Withdrawn by Committee C-11.

Lens.—Withdrawn by Committee E-4.

Load-Bearing Tile.—Withdrawn by Committee C-10 and resubmitted as a definition for "load-bearing wall tile."

Masons' Hydrated Lime.—Withdrawn by Committee C-7.

Misweaves.—Withdrawn by Committee D-13.

Muntz Metal.—Withdrawn by Committee E-4 and incorporated in note under "beta brass."

Planimetric Method for Grain Count.—Withdrawn by Committee E-4 and resubmitted as a note under "grain size."

Recovery.—(D 2) Classified by Committee E-8 as not a definition.

Residue.—(D 2) Classified by Committee E-8 as not a definition.

Saponification Number.—(D-2) Classified by Committee E-8 as not a definition.

Terra Cotta.—Withdrawn by Committee C-10.

Thin Strip.—Withdrawn by Committee D-13.

Virgin Lead.—Withdrawn by Committee B-2 and resubmitted as a section on scope.

Viscosity.—(D-2) Classified by Committee E-8 as not a definition.

Vitrified Tile.—Withdrawn by Committee C-10.

Voids.—Withdrawn by Committee D-1.

RÉSUMÉ OF SUB-COMMITTEE AND OTHER ACTIVITIES

Sub-Committee on Specific Gravity.—A comprehensive study made by this sub-committee under the chairmanship of F. M. Farmer was published with the report of Committee E-8 in 1923. This report received considerable discussion in the several standing committees of the Society and resulted in a report being presented in 1924 by Committee D-4 on Road and Paving Materials. This report was endorsed by Committee D-8 on Waterproofing Materials and Committee D-7 on Timber. The chairman of the sub-committee prepared an analysis of this report which was appended to the report of Committee E-8 in 1924. The chairman of the sub-committee conferred with a special committee of Committee D-4 and communicated with Committees D-2, D-4, D-7 and D-8, the several committees that had submitted comments upon the earlier reports and recommendations of the sub-committee. A compromise agreement on the form that the definitions of specific gravity should take was reached and the sub-committee presented definitions of terms relating to specific gravity which are appended hereto[1] for submission to the Society for publication as tentative. The submission of these definitions has been referred to letter ballot of Committee E-8, the results of the letter ballot vote being as follows: Advisory Committee, 4 affirmative, 1 negative, 2 not voting, total 7. Standing Committees, 23 affirmative, 0 negative, 15 not voting, total 38.

It is to be noted that the number of definitions has been confined to four. It is further to be noted that a definition for "apparent" specific gravity has been retained as definition No. 3. It was thought that the addition of a footnote under definition No. 3 might take care of objections that had been raised to the use of "apparent" in this connection. One of the principal objections that had been raised to previous recommendations of the sub-committee was that no distinction had been contemplated between specific gravity values when the temperature is the same for the material and the water and where it is different. This distinction is now brought out by the use of two forms of expression for specific gravity. The simpler expression for the case where the two temperatures are the same will, it is thought, tend to encourage the practice of using the same temperature and ultimately permit the requirement in the definition itself that the temperatures be the same.

Sub-Committee on Sand and Aggregate (L. R. Ferguson, Chairman).—On the recommendation of this sub-committee a definition of the term "sand" was presented for publication as tentative in 1924, after its publication as information during the year previous. This definition received a review by the Editorial Committee during the past year. In the light of the comments of the Editorial Committee, the sub-committee is now recommending that the Tentative Definition of the Term Sand (C 58 - 24 T)[1] be changed from its present form: namely,

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[1] See p. 887.—ED.

> "*Sand.*—The fine granular material naturally or artificially produced by the disintegration or crushing of rock or slag.
>
> "NOTE.—When used without a qualifying adjective it is generally understood to mean a product of the natural disintegration of siliceous or calcareous rock. The size of particle and other physical characteristics should properly be taken care of in specifications and therefore no limits are mentioned in the definition."

to read as follows:

> "*Sand.*—The fine granular material resulting from the natural disintegration or the crushing of rock or slag.
>
> "NOTE.—When used without a qualifying adjective it is generally understood to mean the product of the natural disintegration of siliceous or calcareous rock. The size of particle and other physical characteristics should be taken care of in specifications and therefore no limits are mentioned in the definition."

This recommendation has been approved by Committee E-8 and has been referred to letter ballot with the following result: Advisory Committee, 4 affirmative, 0 negative, 3 not voting, total 7. Standing Committees, 17 affirmative, 2 negative, 19 not voting, total 38.

The sub-committee is presenting for publication as information a definition of the term "aggregate" reading as follows:

> "*Aggregate.*—Inert mineral material which when bound together into an agglomerated mass by a matrix forms concrete, mastic, mortar, plaster, etc."

Sub-Committee on Moisture, Ash, Volatile Matter and Fixed Carbon.—The chairman of the sub-committee, H. C. Porter, called the attention of Committee E-8 to the fact that the term "moisture" concerns intimately such materials as coal, coke, wood, textile fabrics, paper, oils, starch, flour and many food products, pigments, clays, etc. He recommended that the sub-committee be suitably enlarged to include the representatives of those interested committees that were not represented on this sub-committee. It was accordingly decided to communicate with the various standing committees interested advising them of the existence of the Sub-Committee on Moisture, Ash, Volatile Matter and Fixed Carbon and offering the standing committees representation on the sub-committee. The personnel of the sub-committee has since been enlarged to include the representatives of Committees D-7 on Timber and D-13 on Textile Materials, the complete personnel being as follows:

D-5	H. C. Porter (Chairman)	D-7	Ernest Bateman
D-1	G. B. Heckel	D-13	W. F. Edwards
D-2	F. W. Lane		

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 851 (1924); also 1924 Book of A.S.T.M. Tentative Standards, p. 222.

Sub-Committee on Definitions of the Terms Sieve and Screen.—In the report of Committee E-8 for 1924 a definition for the term "screen (sieve)" was published as information. A number of comments have been received on this definition including certain suggestions by the Editorial Committee. The Sub-Committee on Sieve and Screen, under the chairmanship of L. R. Ferguson, has given consideration to these comments and is now recommending that the following definition for the term "screen (sieve)" be published as a tentative standard:

> "*Screen (Sieve).*—A plate or sheet or a woven cloth, with regularly spaced apertures of uniform size, mounted on a suitable frame or holder, for use in separating materials according to size.
>
> "NOTE.—The shape and spacing of apertures, size of wires or threads, thickness of plate or sheet, allowable variations and similar properties, should be taken care of in specifications."

This recommendation has been approved by Committee E-8 and has been referred to letter ballot with the following results: Advisory Committee, 5 affirmative, 0 negative, 2 not voting, total 7. Standing Committees, 20 affirmative, 0 negative, 18 not voting, total 38.

Definitions of Terms Relating to Timber.—An extensive study of all the definitions of terms relating to timber is being made by Committee D-7 in cooperation with the Central Committee on Lumber Standards. A number of definitions prepared by Committee D-7 were referred to Committee E-8 during the past year. A number of editorial changes have been suggested in these definitions and have been transmitted to Committee D-7. A report will probably be forthcoming during the year on the actions taken by the Central Committee on Lumber Standards on definitions relating to timber with appropriate recommendations by Committee D-7 on these actions.

Definitions of Terms Relating to Heat Treatment.—The American Society for Steel Treating prepared a series of terms relating to heat treatment which were referred to a number of organizations for criticism, including the A.S.T.M. The Executive Committee referred these definitions to Committee A-4 on Heat Treatment of Iron and Steel. Committee A-4 has prepared a number of comments which have been forwarded to the Executive Committee for transmission to the A.S.S.T. It further recommended that a Joint Committee of nine be appointed to consist of three representatives from each of the following organizations: The American Society for Steel Treating, Society of Automotive Engineers and the American Society for Testing Materials.

This report has been submitted to letter ballot of the committee with the following results: Advisory Committee, 5 affirmative, 0 negative, 2 not voting, total 7. Standing Committees, 27 affirmative, 0 negative, 11 not voting, total 38.

Respectfully submitted on behalf of the committee,

CLOYD M. CHAPMAN,
Chairman.

C. L. WARWICK,
Ex-Officio Secretary.

EDITORIAL NOTE

The proposed revision of the Tentative Definition of the Term Sand was accepted. The tentative definition as thus revised appears on page 635.

The proposed new Definitions of Terms Relating to Specific Gravity; and of the Term Screen (Sieve) were accepted for publication as tentative and appear on pages 887 and 889, respectively.

REPORT OF COMMITTEE E-9
ON
CORRELATION OF RESEARCH

At the 1924 annual meeting of the Society, the Executive Committee announced that it had authorized the formation of a committee to be known as the "Committee on Correlation of Research." The formation of this committee was the result of the steady growth of an opinion that the Society should take some specific step looking towards the carrying out of the task of extending the field of knowledge of engineering materials, in addition to the task of formulating specifications and methods of test for materials, to which it has given much time and very successful effort. Suggestions for the initial activities of the new committee were made by the Executive Committee.[1] Accordingly Committee E-9 on Correlation of Research has been organized, with the following personnel:

F. G. Breyer
G. K. Burgess
G. H. Clamer
H. F. Moore (*Chairman*)
F. E. Schmitt
C. L. Warwick (*Ex-officio Secretary*)

The committee has held three meetings: for organization at Atlantic City, June 25, 1924; at Washington, December 30, 1924; and at Philadelphia, April 15, 1925.

The Work of the Committee During the Year

In creating Committee E-9, the Executive Committee suggested that one of its first activities might well be the enumeration and study of the present agencies in the Society for research. Broadly speaking, these are: (1) Standing committees; (2) joint activities with other bodies, either as joint committees or representation of our Society on committees of other bodies, on research organizations, and the like; (3) individual and company members, whose investigations are presented in the form of technical papers and discussions; and (4) the dissemination of knowledge through our publications. The committee has made a preliminary analysis of these four agencies. By far the most extensive of these, and the most important from the present standpoint, is the first. The second is definitely known and is a matter of record in the Secretary-Treasurer's office; while the last two are matters for later consideration. Accordingly, the committee took up as its first major work a survey of such present activities of standing committees of the Society as might be classified under the head of research. This has been made by the secretary of the committee. In making this survey it was found convenient to note three types of activity, each of which may be classed as research: (1) the general study of properties of materials without any direct attempt to use the knowledge gained in the formulation of specifications; (2) study of properties of materials in connection with the work of preparing specifications; and (3) study of methods of testing materials. The latter activity is one which is characteristic of no small part of the work of our Society.

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[1] For further details of the recommendations of the Executive Committee, see the 1924 report *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 58 (1924).

The survey is embodied in a document entitled "List of Current and Proposed Researches and Investigations by A.S.T.M. Committees." It comprises 20 single-spaced typewritten pages and has been prepared in mimeographed form. The List is based upon an examination of standing committee reports from 1919 to 1924, and is intended to include all investigations that are designed primarily to promote the knowledge of engineering materials. Each project of this kind that is definite and has a purpose that can be definitely stated has been given a number and title, and information regarding it is given under three headings: project, administration and status. The List does not include such relatively minor investigations as relate, for example, to some disputed point in a specification and have no further general significance. In addition, there are a number of committees that are devoting their major attention to methods of testing and whose investigative work has necessarily been treated in a general way, the scope of such work having been stated but no attempt made to list the various projects formally.

Copies of this List of Researches have been sent to the chairmen and secretaries of the standing committees of the Society for their review and comment, and a few copies are available for distribution to interested members.[1] Considerable comment has already been received from a number of committees. The research projects are being listed in card index form at the office of the secretary, arranged both as to subject matter and by committees, and in such a way that the records may be kept up to date. This index will also include all researches carried out in cooperation with other societies.

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[1] It is not feasible at the present time to publish the complete text of this List in the Proceedings of the Society. —ED.

In the opinion of Committee E-9 the survey that has been made indicates that there is already going on in the Society a considerable amount of worthwhile research work on the properties of materials, and on the development of methods for determining such properties. The work of nearly all A.S.T.M. committees is characterized by a healthy spirit of constructive criticism and of inquiry and not at all by a feeling that it is either needless or useless to search for more nearly fundamental facts concerning materials.

The following list of pieces of research work already in active progress in connection with the work of various committees of the Society is offered by way of illustration. This list is by no means comprehensive,—in fact, it has been picked in a random way from the much larger general List.

Atmospheric corrosion and water corrosion of sheet steel, both bare and with metallic coatings; including a study of accelerated corrosion tests.
Methods of corrosion-resistance testing.
Tests of various metal screen wire cloths.
Magnetic properties of steel and their correlation with other properties.
Properties of white metal bearing alloys.
Compressive strength tests of cement mortars.
Development of chemical tests for resistance of drain tile to the action of acids and alkalies in soils.
Theory of proportioning and design of concrete.
Studies of properties of varnish.
Study of anti-corrosive and anti-fouling paints.
Study of tests for wide variety of petroleum products.
Study of transformer oils.

In addition to preparing the above-mentioned List of Researches, Committee E-9 has taken action in several matters connected with research in the Society. One of the most important of these is the recommendation that has been made to the Executive Committee for the establishment of an annual research lecture to be delivered each year by some outstanding man in the field of materials. The committee has also recommended that there be awarded annually a medal for a paper representing an outstanding achievement in the field of materials. Both of these recommendations have been approved in principle by the Executive Committee of the Society. Committee E-9 believes that the lecture and the medal will serve the purpose of stimulating research in materials and of keeping before the members the importance of this side of our work, and will have a far-reaching influence in the development of the Society's activities along these lines.

Committee E-9 has recommended the appointment of a research committee on the effect of tin upon high-speed tool steel. This project was brought to the committee by Committee A-9 on Ferro-Alloys as one that enters into certain phases of its work in the preparation of specifications for ferro-tungsten, used largely in the manufacture of tool steel. The matter is one of considerable economic importance because of the constantly decreasing supply of tin-free tungsten ores. A separate small research committee is best able to study this problem. The Executive Committee has approved this recommendation and the committee, when appointed, will be the first research committee of the Society.

Committee A-2 on Wrought Iron asked the advice of Committee E-9 concerning certain research problems in connection with wrought iron. Committee E-9 has recommended that these problems be taken up by Committee A-2 in cooperation with Committee A-4 on Heat Treatment of Iron and Steel, since problems of heat treatment are involved.

When Committee E-9 came into existence, plans were well under way for the formation of a joint committee of the A.S.T.M. and the American Society of Mechanical Engineers to consider the Effect of Temperature on the Properties of Metals. Committee E-9 recommended that this joint committee be formed and has encouraged and followed its work.

THE PLANS OF THE COMMITTEE

The committee plans to keep up from year to year the survey of research work in progress in the various committees of the Society and in cooperation with other bodies, and to study and classify all such researches and investigations.

The committee feels that the matter of corrosion-resistant, heat-resistant and electrical-resistance alloys, which formed the subject matter for an important symposium during the 1924 meeting, should be kept alive and is now in consultation with the committee which developed that symposium as to the best method of bringing this about.

There has recently come to the attention of Committee E-9 a suggestion that valuable results might be obtained from a research on the nature, determination and significance of the yield point of various grades of structural steel. The committee has asked a small committee consisting of representatives of the steel makers, the steel users and testing engineers, to consider this matter and to report whether such a research would be worth while, and if so along what general lines it might be conducted.

During the coming year the committee will inaugurate a survey of the field of engineering materials with a view of determining what important knowledge of certain materials may be required for which research projects may properly be undertaken in some way by the Society. In this connection the standing committees have been asked to advise Committee E-9 of any problems relating to properties of materials they may encounter in their work that require special investigation. The committee now has before it for consideration a number of such problems that have been proposed from various sources, and will welcome suggestions from any member of the Society.

One of the members of the committee, Mr. Breyer, has undertaken the development of an outline of the properties and behavior under various conditions that must be known of any material before it can be said that our knowledge of the material is complete. If such an outline can be prepared it will be of great service in aiding the planning of research work with any material. A tentative outline has been submitted and the committee has asked Mr. Breyer to elaborate it for further study by the committee.

Financial problems are not the major problems confronting Committee E-9, yet the question of financial support of research projects is bound to be vitally important, and the committee in considering any correlation of research work endeavors to give the financial problems involved due consideration and to keep in touch with the committees more directly concerned with the financial interests of the Society.

Respectfully submitted on behalf of the committee,

H. F. MOORE,
Chairman.

C. L. WARWICK,
Secretary.

A.S.T.M.
TENTATIVE STANDARDS

SUBMITTED OR REVISED
AT THE
1925 ANNUAL MEETING.

The term Tentative Standard is applied to a proposed Standard which is printed for one or more years with a view of eliciting criticism, of which the committee concerned will take due cognizance before recommending final action towards its adoption as standard.

Members of the Society and others are invited to direct written criticism of any of these Tentative Standards to the officer of the appropriate committee whose name and address appear in the footnote to the title of each Tentative Standard.

TENTATIVE SPECIFICATIONS
FOR
CARBON-STEEL CAR AND TENDER AXLES[1]

Serial Designation: A 21 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

Scope.

1. These specifications cover tapered axles up to and including those $6\frac{1}{2}$ in. in nominal diameter at the center. Axles over $6\frac{1}{2}$ in. in diameter at the center shall be purchased under the Standard Specifications for Carbon-Steel Forgings for Locomotives (Serial Designation: A 20) of the American Society for Testing Materials.[2]

I. MANUFACTURE

Process.

2. The steel shall be made by the open-hearth or electric-furnace process.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition

3. The steel shall conform to the following requirements as to chemical composition:

Carbon	0.40 - 0.55	per cent
Manganese	0.40 - 0.70	"
Phosphorus, not over	0.05	"
Sulfur, not over	0.06	"

Ladle Analyses.

4. An analysis shall be made by the manufacturer from a test ingot taken during the pouring of each melt, to determine the percentage of carbon, manganese, phosphorus, sulfur and, when specified, silicon. Drillings for analysis shall be taken not less than $\frac{1}{4}$ in. beneath the surface of the test ingot. A copy of this analysis shall be given to the purchaser or his representative. This analysis shall conform to the requirements specified in Section 3.

Check Analyses.

5. Analyses may be made by the purchaser from one axle representing each melt. The chemical composition thus determined shall conform to the requirements specified in Section 3. The drillings for these analyses shall be from one end of the test axle at any point midway between the center and surface, using a $\frac{5}{8}$-in. drill.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

These Tentative Specifications, when adopted as standard, will supersede the present Standard Specifications for Carbon-Steel Car and Tender Axles (Serial Designation: A 21 - 18), 1924 Book of A.S.T.M. Standards.

[2] 1924 Book of A.S.T.M. Standards.

III. PHYSICAL PROPERTIES AND TESTS

Drop Tests. 6. (*a*) The test axle shall be so placed on supports 3 ft. apart, that the tup will strike it midway between the ends. It shall stand without fracture five blows from a tup of 2240 lb. falling from a height H, such that H in feet equals the square of the diameter of the axle at the center in inches, $H=d^2$. The axle shall be turned through 180 deg. after the first and third blows.

(*b*) Before an axle is drop tested, the center shall be calipered to the nearest $\frac{1}{8}$ in., and the height of drop in feet, to the nearest $\frac{1}{2}$ ft., shall not be less than the square of the actual diameter, at the center, in inches.

(*c*) The permanent set produced by the first blow shall not exceed that given by the following formulas, in which L = length of axle in inches, and d = diameter of axle at center in inches.

For axles over 65 in. in length:

$$\left(\frac{L}{1.9d}-\frac{d}{2}\right)+\frac{1}{2}\text{ in} \quad\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

For axles 65 in. or under in length:

$$\left(\frac{L}{1.9d}-\frac{d}{2}\right)+1\text{ in} \quad\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (2)$$

(*d*) The American Railway Association has adopted five standard sizes of axles, the requirements for which, based on the above formulas, are given in the following table:

TABLE I.

Classification of Axles	Size of Journal, in.	Diameter of Axle at Center, in.	Length of Axle, in.	Height of Drop, ft.	Number of Blows	Maximum Permanent Set, in.
A....................	$3\frac{3}{4}$ by 7	$4\frac{1}{4}$ $4\frac{3}{8}$ $4\frac{1}{2}$	$83\frac{1}{4}$	18 19 20	5	$8\frac{3}{4}$ $8\frac{3}{8}$ 8
B....................	$4\frac{1}{4}$ by 8	$4\frac{3}{4}$ $4\frac{7}{8}$ 5	$84\frac{1}{4}$	$22\frac{1}{2}$ $23\frac{1}{2}$ 25	5	$7\frac{1}{2}$ $7\frac{1}{4}$ 7
C....................	5 by 9	$5\frac{3}{8}$ $5\frac{1}{2}$ $5\frac{5}{8}$	$86\frac{1}{2}$	29 30 $31\frac{1}{2}$	5	$6\frac{1}{4}$ 6 $5\frac{3}{4}$
D....................	$5\frac{1}{2}$ by 10	$5\frac{7}{8}$ 6 $6\frac{1}{8}$	$88\frac{1}{2}$	$34\frac{1}{2}$ 36 $37\frac{1}{2}$	5	$5\frac{1}{2}$ $5\frac{1}{4}$ 5
E....................	6 by 11	$6\frac{7}{16}$ $6\frac{9}{16}$ $6\frac{11}{16}$	$90\frac{3}{4}$	$41\frac{1}{2}$ 43 $44\frac{1}{2}$	5	$4\frac{3}{4}$ $4\frac{1}{2}$ $4\frac{1}{4}$

(*e*) The maximum permanent set is the difference between the distance from a straight edge to the middle point of the axle, measured before the first blow, and the distance measured in the same manner after the blow. The straight edge shall rest only on the end collars or the ends of the axle.

(*f*) The temperature of the test axle shall be between 40 and 120° F. (4 and 50° C.).

Drop Test Machine.

7. The anvil of the drop-test machine shall be supported on 12 springs, as shown on the A.R.A. drawings, and shall be free to move in a vertical direction, and shall weigh 17,500 lb. The radii of the striking face of the tup and of the supports shall be 5 in.

Number of Tests.

8. One drop test shall be made from each melt. Not less than 15 tons shall be offered from any one melt unless otherwise specified.

IV. WORKMANSHIP AND FINISH

9. (*a*) All axles shall be made and finished in a workmanlike manner and shall conform to the sizes and shaped specified by the customer and all journals and wheel seats shall be rough turned. In centering, unless otherwise specified, 60-deg. centers shall be used, with large diameter of counter-sink not less than $\frac{7}{8}$ in. and with clearance drilled $\frac{1}{2}$ in. deep.

(*b*) Unless otherwise specified, axles shall be smooth forged, except the wheel seats and journals, which shall be rough turned.

Finish.

10. The axles shall be free from injurious defects and shall have a workmanlike finish.

V. PERMISSIBLE VARIATIONS AND WEIGHTS

Permissible Variations.

11. (*a*) The length of the axles shall not be less than that specified and not more than $\frac{1}{8}$ in. over.

(*b*) *Smooth Forged Axles.*—The diameters of the straight center portion of the axles and the black collars shall not be less than that specified, and shall not be more than $\frac{1}{4}$ in. over. The intermediate portion of the axles shall not be under the dimensions specified, and shall be forged to a straight taper in a workmanlike manner.

(*c*) *Rough Turned Axles.*—The diameters of the straight center portion and the black collars shall not be less than that specified. The straight center portion shall not be more than $\frac{1}{8}$ in. over, and the black collars not more than $\frac{1}{4}$ in. over the diameter specified. The intermediate portion of the axles shall not be under the dimensions specified, and shall be turned to a straight taper in a workmanlike manner.

(*d*) The rough-turned diameter of the journals and wheel seats shall be from ⅛ to ¼ in. over the finished sizes to provide for proper finishing.

Weights. 12. (*a*) The maximum weights of smooth-forged and rough-turned axles given in Section 6 (*d*) in any shipment shall not exceed those given in Table II.

TABLE II.

Classification of Axles	Size of Journal, in.	Maximum Weight, lb.	
		Smooth Forged with Rough Turned Journals and Wheel Seats	Rough Turned All Over
A	3¾ by 7	435	425
B	4¼ by 8	530	520
C	5 by 9	705	695
D	5½ by 10	835	825
E	6 by 11	1015	1005

(*b*) Axles shown in Table II shall be billed at actual weight except that all weight in excess of the above maximum figures shall be at the expense of the manufacturer.

VI. MARKING

Marking. 13. Each axle shall have the manufacturer's name or brand legibly stamped on one of the black collars, and the melt number, the month and year when made stamped on the other, unless otherwise specified.

VII. INSPECTION AND REJECTION

Inspection. 14. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the axles ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the axles are being furnished in accordance with these specifications. Tests and inspection at the place of manufacture shall be made prior to shipment.

(*b*) The purchaser may make the chemical tests to govern the acceptance or rejection of the axles in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

(*c*) All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

15. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 14 (*b*) shall be reported within five working days from the receipt of samples. **Rejection.**

(*b*) Axles which show injurious defects while being finished by the purchaser will be rejected, and the manufacturer shall be notified.

16. Samples tested in accordance with Section 14 (*b*), which represent rejected axles, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time. **Rehearing.**

TENTATIVE SPECIFICATIONS
FOR
STRUCTURAL SILICON STEEL[1]

Serial Designation: A 94 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1925.

Material Covered. 1. These specifications cover a special high-strength structural steel intended primarily for use as main stress-carrying material of structural members.

I. MANUFACTURE

Process. 2. The steel shall be made by the open hearth-process.

Discard. 3. A sufficient discard shall be made from each ingot to secure freedom from injurious piping and undue segregation.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition. 4. The steel shall conform to the following requirements as to chemical composition:

		LADLE ANALYSIS	CHECK ANALYSIS
Carbon, per cent		not over 0.40	not over 0.44
Phosphorus, per cent	Acid	" " 0.06	" " 0.075
	Basic	" " 0.04	" " 0.05
Sulfur, per cent		" " 0.05	" " 0.063
Silicon, per cent		" under 0.20	" under 0.18

Ladle Analyses. 5. An analysis of each melt of steel shall be made by the manufacturer to determine the percentages of carbon, manganese, phosphorus, sulfur and silicon. This analysis shall be made from a test ingot taken during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative and shall conform to the requirements specified in Section 4 for ladle analysis.

Check Analyses. 6. Analyses may be made by the purchaser from finished material representing each melt. The chemical composition thus determined shall conform to the requirements specified in Section 4 for check analysis.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

III. PHYSICAL PROPERTIES AND TESTS

Tension Tests.

7. (*a*) The material shall conform to the following requirements as to tensile properties:

Tensile strength, lb. per sq. in.	80 000 – 95 000
Yield point, min., lb. per sq. in.	45 000
Elongation in 8 in., min., per cent	$\frac{1\,500\,000^{a}}{\text{tens. str.}}$
Reduction of area, min., per cent	30[b]

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[a] See Section 8.

[b] See Section 9.

(*b*) The yield point shall be determined by the drop of the beam of the testing machine when operated at a cross-head speed not greater than ½ in. per minute. The testing machine shall not be stopped to obtain the drop of the beam.

Modifications in Elongation.

8. For material over ¾ in. in thickness, a deduction from the percentage of elongation specified in Section 7 (*a*) of 0.25 per cent shall be made for each increase of $\frac{1}{16}$ in. of the specified thickness above ¾ in., to a minimum of 14 per cent.

Modifications in Reduction of Area.

9. For material over ¾ in. in thickness, a deduction from the percentage of reduction of area specified in Section 7 (*a*) of 0.50 per cent shall be made for each increase of $\frac{1}{16}$ in. of the specified thickness above ¾ in., to a minimum of 24 per cent.

Bend Tests.

10. The test specimen shall stand being bent cold through 180 deg. without cracking on the outside of the bent portion around a pin, the diameter of which shall have the following relation to the thickness of the specimen:

SPECIMEN THICKNESS	PIN DIAMETER
Up to ¾ in., inclusive	1 times the thickness
Over ¾ in. to 1 in., inclusive	1½ " " "
Over 1 in. to 1¼ in., inclusive	2 " " "
Over 1¼ in.	2½ " " "

Test Specimens.

11. (*a*) Test specimens shall be prepared for testing from the material in its rolled or forged condition, except as specified in Paragraph (*b*).

(*b*) Test specimens for annealed material shall be prepared from the material as annealed for use, or from a short length of a full section similarly treated.

(*c*) Test specimens shall be taken longitudinally, and, except as specified in Paragraphs (*e*) and (*f*), shall be of the full thickness or section of material as rolled.

(*d*) Test specimens for plates, shapes and flats may be machined to the form and dimensions shown in Fig. 1, or with both edges parallel.

(*e*) Tension test specimens for material over 1 in. in thickness or diameter may be machined to a thickness or diameter of at least $\frac{3}{4}$ in., for a length of at least 9 in.

(*f*) Bend test specimens for material over $1\frac{1}{4}$ in. in thickness or diameter may be machined to a thickness or diameter of at least $\frac{3}{4}$ in. or to 1 by $\frac{1}{2}$ in. in section.

(*g*) The machined sides of rectangular bend test specimens may have the corners rounded to a radius not over $\frac{1}{16}$ in.

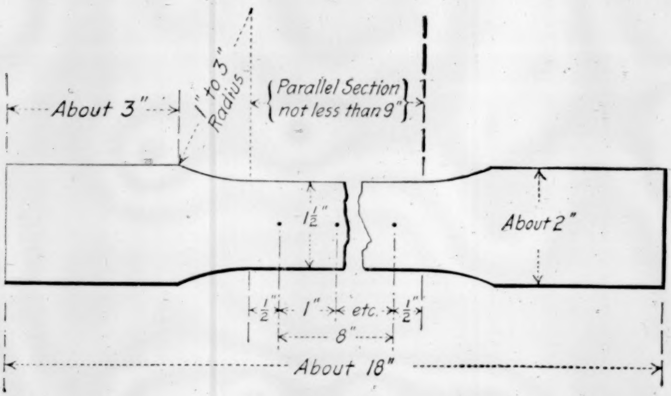

FIG. 1.

Number of Tests.

12. (*a*) Two tension and one bend test shall be made from each melt; except that if material from one melt differs $\frac{3}{8}$ in. or more in thickness, one tension and one bend test shall be made from both the thickest and the thinnest material rolled.

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 7 (*a*) and any part of the fracture is outside the middle third of the gage length, as indicated by marks placed on the specimen before testing, a retest shall be allowed.

IV. PERMISSIBLE VARIATIONS IN WEIGHT AND THICKNESS

Permissible Variations.

13. The cross-section or weight of each piece of steel shall not vary more than 2.5 per cent from that specified; except in the case of sheared plates, which shall be covered by the following permissible variations. One cubic inch of rolled steel is assumed to weigh 0.2833 lb.

(*a*) *When Ordered to Weight per Square Foot:* The weight of each lot[1] in each shipment shall not vary from the weight ordered more than the amount given in Table I.

TABLE I.—PERMISSIBLE VARIATIONS OF RECTANGULAR PLATES ORDERED TO WEIGHT.

Ordered Weight, lb. per sq. ft.	Permissible Variations in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Ordered Weights																		Ordered Weight, lb. per sq. ft.
	Under 48 in.		48 to 60 in., excl.		60 to 72 in., excl.		72 to 84 in., excl.		84 to 96 in., excl.		96 to 108 in., excl.		108 to 120 in., excl.		120 to 132 in., excl.		132 in. or over		
	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	
Under 5........	5	3	5.5	3	6	3	7	3	...	...	...	...	...	...	...	...	...	...	Under 5
5 to 7.5 excl.	4.5	3	5	3	5.5	3	6	3	...	...	...	...	...	...	...	...	...	...	5 to 7.5 excl.
7.5 " 10 "	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	...	...	...	...	7.5 " 10 "
10 " 12.5 "	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	9	3	10 " 12.5 "
12.5 " 15 "	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	12.5 " 15 "
15 " 17.5 "	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	15 " 17.5 "
17.5 " 20 "	2.5	2	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	17.5 " 20 "
20 " 25 "	2	2	2.5	2	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	20 " 25 "
25 " 30 "	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	4.5	3	5	3	25 " 30 "
30 " 40 "	2	2	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	4.5	3	30 " 40 "
40 or over......	2	2	2	2	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	40 or over

NOTE.—The weight per square foot of individual plates shall not vary from the ordered weight by more than $1\frac{1}{3}$ times the amount given in this table.

TABLE II.—PERMISSIBLE OVERWEIGHTS OF RECTANGULAR PLATES ORDERED TO THICKNESS.

Ordered Thickness, in.	Permissible Excess in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Nominal Weights									Ordered Thickness, in.
	Under 48 in.	48 to 60 in., excl.	60 to 72 in. excl.	72 to 84 in., excl.	84 to 96 in., excl.	96 to 108 in., excl.	108 to 120 in., excl.	120 to 132 in., excl.	132 in. or over	
Under $\frac{1}{8}$..........	9	10	12	14	..	..	..	..	..	Under $\frac{1}{8}$
$\frac{1}{8}$ to $\frac{3}{16}$ excl.....	8	9	10	12	..	..	..	..	..	$\frac{1}{8}$ to $\frac{3}{16}$ excl.
$\frac{3}{16}$ " $\frac{1}{4}$ "	7	8	9	10	12	..	..	..	..	$\frac{3}{16}$ " $\frac{1}{4}$ "
$\frac{1}{4}$ " $\frac{5}{16}$ "	6	7	8	9	10	12	14	16	19	$\frac{1}{4}$ " $\frac{5}{16}$ "
$\frac{5}{16}$ " $\frac{3}{8}$ "	5	6	7	8	9	10	12	14	17	$\frac{5}{16}$ " $\frac{3}{8}$ "
$\frac{3}{8}$ " $\frac{7}{16}$ "	4.5	5	6	7	8	9	10	12	15	$\frac{3}{8}$ " $\frac{7}{16}$ "
$\frac{7}{16}$ " $\frac{1}{2}$ "	4	4.5	5	6	7	8	9	10	13	$\frac{7}{16}$ " $\frac{1}{2}$ "
$\frac{1}{2}$ " $\frac{5}{8}$ "	3.5	4	4.5	5	6	7	8	9	11	$\frac{1}{2}$ " $\frac{5}{8}$ "
$\frac{5}{8}$ " $\frac{3}{4}$ "	3	3.5	4	4.5	5	6	7	8	9	$\frac{5}{8}$ " $\frac{3}{4}$ "
$\frac{3}{4}$ " 1 "	2.5	3	3.5	4	4.5	5	6	7	8	$\frac{3}{4}$ " 1 "
1 or over	2.5	2.5	3	3.5	4	4.5	5	6	7	1 or over

(*b*) *When Ordered to Thickness:* The thickness of each plate shall not vary more than 0.01 in. under that ordered.

The overweight of each lot[1] in each shipment shall not exceed the amount given in Table II.

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[1] The term "lot" as applied to Table I means all of the plates of each group width and group weight; as applied to Table II, it means all of the plates of each group width and group thickness.

V. FINISH

Finish. 14. The finished material shall be free from injurious defects and shall have a workmanlike finish.

VI. MARKING

Marking. 15. The name or brand of the manufacturer and the melt number shall be legibly stamped or rolled on all finished material. The melt number shall be legibly marked, by stamping if practicable, on each test specimen.

VII. INSPECTION AND REJECTION

Inspection. 16. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 17. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 6 shall be reported within five working days from the receipt of samples.

(*b*) Material which shows injurious defects subsequent to its acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing. 18. Samples tested in accordance with Section 6, which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS
FOR
CARBON-STEEL CASTINGS FOR VALVES, FLANGES AND FITTINGS FOR HIGH-TEMPERATURE SERVICE[1]

Serial Designation: A 95 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1925.

Material Covered. 1. These specifications cover carbon-steel castings for valves, flanges and fittings for service up to 750° F. (400° C.) total temperature.

I. MANUFACTURE

Process. 2. The steel shall be made by one or more of the following processes: open-hearth, electric-furnace, converter, or crucible.

Heat Treatment. 3. (*a*) All castings shall receive a heat treatment proper to the design and chemical composition of the castings.

(*b*) Heat treatment, unless otherwise specified by the purchaser, may consist of annealing, or of normalizing, or of normalizing followed by annealing, or of normalizing followed by a draw-back to a temperature below the critical range. No castings which have been quenched in any liquid medium shall be offered under these specifications.

(*c*) *Annealing.*—The procedure for annealing shall consist in allowing the castings to cool, after pouring, to a temperature below the critical range. They shall then be uniformly reheated to the proper temperature to refine the grain and allowed to cool uniformly in the furnace.

(*d*) *Normalizing.*—The procedure for normalizing shall consist in allowing the castings to cool, after pouring, to a temperature below the critical range. They shall then be reheated to the proper temperature to refine the grain and allowed to cool in still air.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition 4. (*a*) The castings shall conform to the following requirements as to chemical composition, except that the following limitations for carbon, manganese and silicon are intended to show desired composition and are not to be used for rejection:

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

Carbon	0.15 – 0.40	per cent
Manganese	not under 0.50	"
Silicon	not under 0.20	"
Phosphorus	not over 0.05	"
Sulfur	not over 0.05	"

(*b*) Manufacturers submitting proposals to furnish material under these specifications shall specify, with their proposals, the limits of carbon, manganese and silicon which they will accept as a basis for rejection.

Ladle Analyses.

5. An analysis of each melt of steel shall be made by the manufacturer to determine the percentages of the elements specified in Section 4. This analysis shall be made from drillings taken at least ¼ in. beneath the surface of a test ingot obtained during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative and shall conform to the requirements specified in Section 4.

Check Analyses.

6. Analyses may be made by the purchaser from the broken tension test specimen or from a casting representing each melt. The chemical composition thus determined shall conform to the requirements specified in Section 4. Samples for analysis shall be taken not less than ¼ in. beneath the surface, except in the case of castings which are too thin, and shall be taken in such a manner as not to impair the usefulness of the casting.

III. PHYSICAL PROPERTIES AND TESTS

Tension Tests.

7. (*a*) The castings shall conform to the following minimum requirements as to tensile properties:

Tensile strength, lb. per sq. in	70 000
Yield point, lb. per sq. in	36 000
Elongation in 2 in., per cent	22
Reduction of area, per cent	30

(*b*) The yield point shall be determined by the drop of the beam of the testing machine at a cross-head speed not to exceed ⅛ in. per minute. The tensile strength shall be determined at a speed not to exceed 1½ in. per minute.

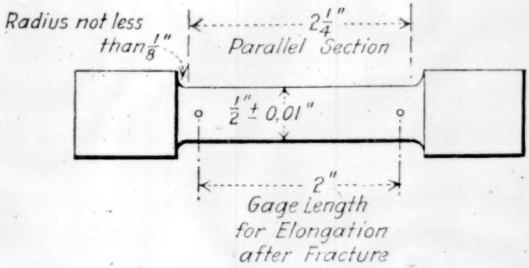
Bend Test.

8. (*a*) Bend tests shall be required for castings only when so specified in the order.

(*b*) When a bend test is specified, the test specimens shall stand being bent cold through an angle of 90 deg. around a pin 1 in. in diameter, without cracking on the outside of the bent portion.

Test Specimens.

9. (*a*) Tension test specimens, and, when a bend test is specified, bend test specimens, shall be taken from test bars cast attached to the castings where practicable. If the design of the castings is such that test bars should not be attached to the castings, the test bars shall be cast attached to special blocks, a sufficient number of which shall be provided for each lot of castings. Test bars from which tension and bend test specimens are to be taken shall remain attached to the castings or blocks they represent through heat-treatment and until presented for inspection. Test bars shall be provided in sufficient numbers to furnish the tests required in Section 10.


Radius not less than 1/8"
2 1/4"
Parallel Section
1/2" ± 0.01"
2"
Gage Length for Elongation after Fracture

Note:-The Gage Length, Parallel Section, and Fillets shall be as Shown, but the Ends may be of any Shape to fit the Holders of the Testing Machine in such a Way that the Load shall be axial.

FIG. 1.

(*b*) If satisfactory to the manufacturer and inspector, tension test specimens may be cut from heat-treated castings, instead of from test bars.

(*c*) Tension test specimens shall conform to the dimensions shown in Fig. 1. The ends shall be of a form to fit the holders of the testing machine in such a way that the load shall be axial.

(*d*) Bend test specimens shall be machined to 1 by ½ in. in section with the corners rounded to a radius of not over $\frac{1}{16}$ in.

Number of Tests.

10. (*a*) One tension test and, when specified, one bend test shall be made from each melt in each heat-treatment charge and, when specified, from each casting weighing 500 lb. or over.

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded; in which case another specimen from the same lot shall be substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 7 (*a*) and any part of the fracture is more than ¾ in. from the center of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Retests. 11. If the results of the physical tests for any lot do not conform to the requirements specified, such lot may be re-treated, but not more than twice. Retests shall be made as specified in Sections 7 and 8.

IV. WORKMANSHIP AND FINISH

Workmanship. 12. The castings shall conform substantially to the shapes and sizes indicated by the patterns and drawings submitted by the purchaser.

Finish. 13. (*a*) The castings shall be free from injurious defects.

Welding. (*b*) Defects which do not impair the strength of the castings may be welded by an approved process. The defects shall be cleaned out to solid metal, before welding, and when so required by the inspector, shall be submitted to him in this condition for his approval. All castings shall be heat-treated after welding in accordance with the requirements in Section 3.

V. INSPECTION AND REJECTION

Inspection. 14. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the castings ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the castings are being furnished in accordance with these specifications.

(*b*) If, in the case of important castings for special purposes, surface inspection in the green state is required, this shall be so specified in the order.

(*c*) All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 15. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 6 shall be reported within five working days from the receipt of samples.

(*b*) Castings which show injurious defects subsequent to their acceptance at the manufacturer's works will be rejected and the manufacturer shall be notified.

Rehearing. 16. Samples tested in accordance with Section 6, which represent rejected castings, shall be preserved for two weeks from the date of transmission of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS
FOR
ALLOY-STEEL BOLTING MATERIAL FOR HIGH-TEMPERATURE SERVICE[1]

Serial Designation: A 96 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925

Material Covered.

1. (*a*) These specifications cover alloy-steel bolting material for valves, flanges and fittings for temperatures from 400 to 750° F. (205 to 400° C.), inclusive. The term "bolting material" as used in these specifications covers rolled, forged or cold-drawn bars, and bolts, screws and studs.

(*b*) Three classes of material are covered, A, B and C, classified in accordance with their physical properties as specified in Section 10.

(*c*) Nickel, chrome-nickel, chrome-vanadium, chrome-manganese or any other types of alloy steel approved by the purchaser may be submitted under these specifications.

Basis of Purchase.

2. When agreed upon between the manufacturer and the purchaser, Brinell hardness tests may be made to determine the acceptance of bolting material in lieu of the tension tests herein specified; but shall not be used as a basis for rejection without confirming tension tests being made.

Certification of Test for Bolts, Screws and Studs.

3. Unless otherwise specified on the order, a certification that bolts, screws and studs for valves and other fittings intended for stock and other purposes requiring assembly in the manufacturer's plant conform to the requirements of these specifications, shall be accepted in lieu of the tests herein specified.

I. MANUFACTURE

Process.

4. The steel shall be made by one or more of the following processes: open-hearth, electric-furnace or crucible.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

Discard. 5. A sufficient discard shall be made from each ingot to secure reedom from injurious piping and undue segregation.

Heat Treatment. 6. (*a*) Heat treatment shall consist of quenching and tempering.

(*b*) Immediately after rolling or forging, the bolting material shall be allowed to cool to a temperature below the critical range under suitable conditions to prevent injury by too rapid cooling. For quenching and tempering, the material shall then be uniformly reheated to the proper temperature to refine the grain (a group thus reheated being known as a "quenching charge") and quenched in some medium under substantially uniform conditions for each quenching charge. The material shall then be uniformly reheated to the proper temperature for tempering or "drawing back" (a group thus reheated being known as a "tempering charge") and allowed to cool uniformly.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition. 7. (*a*) The steel shall conform to the following requirements as to chemical composition:

Phosphorus, per cent	not over 0.04
Sulfur, per cent	not over 0.05

(*b*) The composition of the steel furnished under these specifications, other than phosphorus and sulfur, shall be agreed upon by the manufacturer and the purchaser.

NOTE.—It is recommended that the carbon content shall not be less than 0.20 nor more than 0.45 per cent, and that the carbon ranges shall be 0.10 per cent of carbon.

Ladle Analyses. 8. An analysis of each melt of steel shall be made by the steel manufacturer to determine the percentages of the elements present. This analysis shall be made from a test ingot taken during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 7.

Check Analyses. 9. Analyses may be made by the purchaser from samples representing the bolting material. The chemical composition thus determined shall conform to the requirements specified in Section 7, except that the rejection limit for phosphorus shall be 0.05 per cent and for sulfur 0.055 per cent.

III. PHYSICAL PROPERTIES AND TESTS

Tension Tests. 10. (*a*) The bolting material, after final heat treatment, shall conform to the following requirements as to tensile properties:

	CLASS A	CLASS B	CLASS C
Tensile strength, lb. per sq. in. .	95 000 - 115 000	105 000 - 125 000	not less than 125 000
Yield point, min., lb. per sq. in. .	70 000	80 000	105 000
Elongation in 2 in., min., per cent	20	20	16
Reduction of area, min., per cent	50	50	50

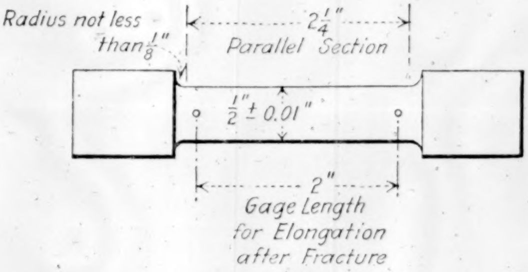
(*b*) The yield point shall be determined by the drop of the beam of the testing machine or by the use of dividers.

Brinell Hardness Tests.

11. (*a*) When Brinell hardness tests are made, the bolting material, after final heat treatment, shall conform to the following requirements:

	BRINELL HARDNESS NUMBER
Class A	190 - 250
Class B	210 - 270
Class C	260 - 320

(*b*) The Brinell hardness test shall be made in accordance with the Tentative Methods of Brinell Hardness Testing of Metallic Materials (Serial Designation: E 10 - 25 T) of the American Society for Testing Materials.[1]


Radius not less than 1/8"
2 1/4"
Parallel Section
1/2" ± 0.01"
2"
Gage Length for Elongation after Fracture

Note:- The Gage Length, Parallel Section, and Fillets shall be as Shown, but the Ends may be of any Shape to fit the Holders of the Testing Machine in such a Way that the Load shall be axial.

FIG. 1.

Tension Test Specimens.

12. (*a*) The tension test specimens taken from the bolting material shall conform to the dimensions shown in Fig. 1, except as specified in Paragraph (*b*). The ends shall be of a form to fit the holders of the testing machine in such a way that the load shall be axial.

(*b*) In the case of small sections which will not permit of taking the standard tension test specimen specified in Paragraph (*a*), the tension test specimen shall be as large as feasible and its dimensions shall be proportional to those shown in Fig. 1. The gage length for measuring elongation shall be four times the diameter of the specimen.[2]

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[1] See p. 872.

[2] These requirements are in accordance with the provisions of Section 5 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 - 25 T), see p. 854.

Number of Tests. 13. (*a*) Except as specified in Paragraph (*b*), one tension test shall be made for each lot of 100 pieces or less of bolting material from each tempering charge. If more than one quenching charge is represented in a tempering charge, one tension test shall be made from each lot of 100 pieces or less from each quenching charge. If more than one melt is represented in a quenching charge, one tension test shall be made from each lot of 100 pieces or less from each melt.

(*b*) Unless required on the order, tension tests shall not be made on an order of less than 100 bolts, screws or studs; in which case acceptance shall be based upon the provisions in Sections 2 or 3.

(*c*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*d*) If the percentage of elongation of any test specimen is less than that specified in Section 10 (*a*) and any part of the fracture is outside of the middle three-fourths of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Retests. 14. If the results of the physical tests of any lot of bolting material do not conform to the requirements specified, the manufacturer may re-treat such lot one or more times and retests shall be made as specified in Section 13 (*a*).

IV. WORKMANSHIP AND FINISH

Finish. 15. The bolting material shall be free from injurious defects and shall have a workmanlike finish.

Nuts and Washers. 16. Bolts and studs shall be equipped with cold-punched semi-finished carbon-steel nuts of U. S. Standard rough dimensions, chamfered and trimmed. If washers are used under nuts they shall be of forged or rolled steel. All nuts and washers shall be free from injurious defects and shall have a workmanlike finish, and shall conform to the chemical requirements for phosphorus and sulfur specified in Sections 7 (*a*) and 9.

Threads. 17. U. S. Standard threads shall be used, except that no stud shall have less than eight threads per inch.

V. MARKING

Identification Marks. 18. Identification marks to be stamped on bolting material shall be agreed upon between the manufacturer and the purchaser.

VI. INSPECTION AND REJECTION

Inspection. 19. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the bolting material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the bolting material is being furnished in accordance with these specifications. Tests and inspection at the place of manufacture shall be made prior to shipment.

(*b*) The purchaser may make the tests to govern the acceptance or rejection of the bolting material in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

(*c*) Tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection.

20. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 19 (*b*) shall be reported within five working days from the receipt of samples.

(*b*) Bolting material which shows injurious defects while being finished by the purchaser will be rejected and the manufacturer shall be notified.

Rehearing.

21. Samples tested in accordance with Section 19 (*b*), which represent rejected bolting material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the test, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS
FOR
TUNGSTEN POWDER[1]

Serial Designation: A 97 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Grades. 1. These specifications cover tungsten powder in one grade only.

Basis of Purchase. 2. The tungsten powder shall be thoroughly mixed and packed in sound containers preferably metallic or metallic lined and sufficiently strong to prevent loss in transportation.

Chemical Requirements. 3. The material shall conform to the following requirements as to chemical composition:

Metallic Tungsten, minimum	95.00	per cent
Carbon, maximum	0.50	"
Sulfur, maximum	0.05	"
Phosphorus, maximum	0.05	"
Oxygen, maximum	1.00	"
Silicon, maximum	0.75	"
Tin, maximum	0.03	"
Copper, maximum	0.03	"
Arsenic, maximum	0.03	"
Bismuth, maximum	0.03	"
Antimony, maximum	0.03	"

Sampling. 4. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 – 25 T) of the American Society for Testing Materials.[2]

Methods of Chemical Analysis. 5. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104 – 25 T) of the American Society for Testing Materials.[3]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 537. [3] See p. 542.

TENTATIVE SPECIFICATIONS
FOR
SPIEGELEISEN[1]

Serial Designation: A 98 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Grades

1. These specifications cover two grades of spiegeleisen, as follows:

Grade A; and
Grade B.

Basis of Purchase.

2. The spiegeleisen shall be furnished in pigs or lumps and each shipment shall be uniform as to the grade specified.

Chemical Requirements.

3. The material shall conform to the following requirements as to chemical composition:

	MANGANESE, PER CENT	CARBON, MAX., PER CENT	PHOSPHORUS, MAX., PER CENT	SULFUR, MAX., PER CENT	SILICON, MAX., PER CENT
Grade A......	19 to 21	6.50	0.15	0.04	as specified
Grade B......	16 to 19	6.50	0.25	0.05	as specified

Sampling.

4. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 - 25 T) of the American Society for Testing Materials.[2]

Methods of Chemical Analysis.

5. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104 - 25 T) of the American Society for Testing Materials.[3]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 537. [3] See p. 542.

TENTATIVE SPECIFICATIONS
FOR
FERRO-MANGANESE[1]

Serial Designation: A 99 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Grades. 1. These specifications cover ferro-manganese in one grade only.

Basis of Purchase. 2. Ferro-manganese shall be furnished in pigs, lumps, crushed or screened to size, as specified.

Chemical Requirements. 3. The material shall conform to the following requirements as to chemical composition:

Manganese, minimum	78.00 per cent
Phosphorus, maximum	0.35 "
Carbon, maximum	7.5 "
Silicon, maximum	1.00 "
Sulfur, maximum	0.050 "

Sampling. 4. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 – 25 T) of the American Society for Testing Materials.[2]

Methods of Chemical Analysis. 5. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104 – 25 T) of the American Society for Testing Materials.[3]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 537. [3] See p. 542.

TENTATIVE SPECIFICATIONS
FOR
FERRO-SILICON[1]

Serial Designation: A 100 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Grades.

1. These specifications cover three grades of ferro-silicon, as follows:

Grade A;
Grade B; and
Grade C.

Basis of Purchase.

2. (*a*) The ferro-silicon shall be furnished in lumps, or crushed or screened to size, as specified.

(*b*) When furnished in lump or granulated form the ferro-silicon shall be free from excessive disintegration.

Chemical Requirements.

3. The material shall conform to the following requirements as to chemical composition:

	GRADE A	GRADE B	GRADE C
Silicon, per cent...........	47.00 to 53.00	72.00 to 78.00	85.00 to 95.00

4. An analysis of each shipment of ferro-silicon shall be furnished the purchaser, showing the percentage of silicon.

Sampling.

5. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 - 25 T) of the American Society for Testing Materials.[2]

Methods of Chemical Analysis.

6. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104 - 25 T) of the American Society for Testing Materials.[3]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 537. [3] See p. 542.

TENTATIVE SPECIFICATIONS
FOR
FERRO-CHROMIUM[1]

Serial Designation: A 101 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Grades.

1. These specifications cover ferro-chromium in four grades, as follows:

High Carbon, Grade A;
Low Carbon, Grades B, C and D.

Basis of Purchase.

2. *Ferro-Chromium, Low Carbon.*—This material shall be crushed to the specified size and mixed before packing, so that the quality in each package is uniform with the lot.

Ferro-Chromium, High-Carbon.—This material shall be furnished to size as specified.

Chemical Requirements.

3. The material shall conform to the following requirements as to chemical composition:

	High Carbon	Low Carbon		
	Grade A	Grade B	Grade C	Grade D
Chromium, per cent	60.00 to 75.00	60.00 to 75.00	60.00 to 75.00	60.00 to 75.00
Carbon, per cent	4.00 to 8.00 as specified	1.50 to 2.00	1.00 to 1.50	under 1.00 as specified[a]
Silicon, per cent	as specified[1]	as specified	as specified	as specified

[a] Grade D material may be obtained with various maxima carbon contents down to 0.10 per cent of carbon.

Sampling.

4. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 – 25 T) of the American Society for Testing Materials.[2]

Methods of Chemical Analysis.

5. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104 – 25 T) of the American Society for Testing Materials.[3]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 537. [3] See p. 542.

TENTATIVE SPECIFICATIONS
FOR
FERRO-VANADIUM[1]

Serial Designation: A 102 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Grades.

1. These specifications cover ferro-vanadium in four grades, as follows:

Grade A;
Grade B;
Grade C; and
Grade D.

Basis of Purchase.

2. The material furnished under this specification shall be crushed to the specified size, and mixed before packing, so that the quality in each package is uniform with the lot. The material shall be packed in sound containers, sufficiently strong to prevent loss in transportation.

Chemical Requirements.

3. The material shall conform to the following requirements as to chemical composition:

	Grade A	Grade B	Grade C	Grade D
Vanadium, per cent	30.00 to 40.00	30.00 to 40.00	35.00 to 45.00	35.00 to 45.00
Carbon, per cent	3.00 to 6.00	1.5 to 3.00	not over 1.5	not over 0.75
Silicon, per cent	8.00 to 15.00	5.00 to 8.00	not over 2.00	not over 2.00
Phosphorus, per cent	not over 0.250	not over 0.250	not over 0.150	not over 0.100
Sulfur, per cent	not over 0.300	not over 0.300	not over 0.200	not over 0.100
Aluminum, per cent	not over 2.00	not over 2.00	not over 2.00	not over 1.00

Sampling.

4. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 - 25 T) of the American Society for Testing Materials.[2]

Methods of Chemical Analysis.

5. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104-25 T) of the American Society for Testing Materials.[3]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 537. [3] See p. 542.

TENTATIVE SPECIFICATIONS
FOR
HOLLOW STAYBOLT IRON.[1]

Serial Designation: A 86 - 25 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1925.

I. MANUFACTURE.

Process.

1. The iron shall be rolled from a bloom, slab pile or box pile, made wholly from reworked puddled pig iron or reworked knobbled charcoal iron. The puddle mixture and the component parts of the bloom, slab pile or box pile shall be free from any admixture of iron scrap or steel.

Definition of Terms.

2. (*a*) *Bloom.*—A bloom is a solid mass of iron that has been hammered into a convenient size for rolling.

(*b*) *Slab pile.*—A slab pile is built up wholly of flat bars of iron of the full length of the pile.

(*c*) *Box pile.*—A box pile is a pile, the sides, top and bottom of which are formed by four flat bars and the interior of which consists of a number of small bars the full length of the pile.

(*d*) *Iron Scrap.*—This term applies only to foreign or bought scrap and does not include local mill products free from foreign or bought scrap.

II. CHEMICAL PROPERTIES AND TESTS.

Chemical Composition.

3. The iron shall conform to the following requirement as to chemical composition:

Manganese	not over 0.10 per cent.

Check Analyses.

4. (*a*) An anlysis may be made by the purchaser from a broken tension test specimen representing each lot as specified in Section 10 (*a*). The chemical composition thus determined shall conform to the requirement specified in Section 3.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. C. C. Osterhout, Secretary of Committee A-2 on Wrought Iron, Rome Iron Mills, Inc., Rome, N. Y.

(*b*) Drillings for analysis shall be so taken as to represent the entire cross-section of the specimen.

III. PHYSICAL PROPERTIES AND TESTS.

Tension Tests. 5. (*a*) The iron shall conform to the following requirements as to tensile properties:

Tensile strength, lb. per sq. in.	48 000 – 52 000
Yield point, min., lb. per sq. in.	0.6 tens. str.
Elongation, when measured in 8 in., min., per cent...	28
Elongation, " " " 4 " " " ...	35
Reduction of area, min., per cent..................	42

(*b*) The yield point shall be determined by the drop of the beam of the testing machine. The speed of the cross-head of the machine shall not exceed $\frac{3}{4}$ in. per minute.

(*c*) In calculating the tensile strength and elastic limit, the area of a $\frac{7}{32}$-in. hole shall be deducted from the area of the bar, but the area of the hole shall be disregarded in calculating the reduction of area.

Bend Tests. 6. (*a*) The test specimen shall withstand being bent cold through 180 deg. flat on itself in both directions, without fracture on the outside of the bent portion.

(*b*) Bend tests may be made by pressure or by blows.

Splitting Tests. 7. A piece not less than 2 in. long shall be split open from end to end by driving a drift or wedge through the hole. The structure thus exhibited shall be free from signs of imperfect welding and the presence of slag or scale in the welds.

Etch Tests. 8. The cross-section of the test specimen shall be ground or polished, and etched[1] for a sufficient period to develop the structure. This test shall show the material to have been rolled from a bloom, slab pile or box pile, and to be free from steel.

Test Specimens. 9. All test specimens shall be of the full section of material as rolled.

Number of Tests. 10. (*a*) Bars of one size shall be sorted into lots of 1600 lineal feet. Two samples shall be selected at random from each lot and tested as specified in Sections 5, 6 and 7; but only one of these shall be tested as specified in Sections 4 and 8.

(*b*) If any of the samples originally selected to represent a lot of material contains surface defects not visible before testing but visible after testing, or if a tension test specimen breaks outside the middle third of the gage length, the individual sample shall be rejected and one retest from a different bar or bolt will be allowed.

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[1] A solution of 10 per cent hydrochloric acid (sp. gr. 1.19), 30 per cent sulfuric acid (sp. gr. 1.84), and 60 per cent water; or 25 per cent nitric acid (sp. gr. 1.42), and 75 per cent water, is recommended for the etch test.

IV. PERMISSIBLE VARIATIONS IN GAGE.

Permissible Variations. 11. The bars shall be truly round within 0.01 in. and shall be not less than 0.005 in. over, nor more than 0.020 in. over the specified diameter.

V. FINISH.

Finish. 12. The bars shall be smoothly rolled and free from slivers, depressions, seams, crop ends, and evidences of being burnt.

VI. MARKING.

Marking. 13. The material shall be suitably marked for identification.

VII. INSPECTION AND REJECTION.

Inspection. 14. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, free of charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. Tests and inspection at the place of manufacture shall be made prior to shipment.

(*b*) The purchaser may make the tests to govern the acceptance or rejection of material in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

(*c*) All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 15. (*a*) If either of the samples selected to represent a lot does not conform to the requirements specified in Sections 3, 5, 6, 7, 8 and 9, the lot will be rejected.

(*b*) Unless otherwise specified, any rejection based on tests made in accordance with Section 14 (*b*) shall be reported within five working days from the receipt of samples.

(*c*) Bars which will not take a clean, sharp thread with dies in fair condition, or which develop defects in forging or machining, will be rejected, and the manufacturer shall be notified.

Rehearing. 16. Samples tested in accordance with Section 14 (*b*), which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE METHODS
OF
SAMPLING FERRO-ALLOYS[1]

Serial Designation: A 103 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

The procedures recommended in these methods are considerably more elaborate than those in general use by the principal consumers of ferro-alloys in this country. However, the methods have been designed as standard procedure for use in important cases where large sums are often involved and they accordingly should represent the most approved modern practice.

FERRO-SILICON, HIGH-CARBON FERRO-MANGANESE AND SILICO-MANGANESE

These alloys are usually shipped in bulk in carloads and should be sampled while being loaded or unloaded. At these times the sampler can make the fairest estimate of the ratio of lump to fine material and should secure proportionate amounts of each. Frequently the percentage of fines is negligible.

While the shipment is being loaded or unloaded, samples aggregating 150 lb. or more shall be gathered from all parts of the car with due regard to the estimated ratio of lump to fine material. In breaking lumps or pigs, fragments representing the full cross-section should be secured, a heavy hammer or sledge being employed.

The gross sample thus obtained shall be crushed to pea size in a heavy crusher provided with smooth plates of manganese steel. The crushed product shall be repeatedly mixed and halved with a Jones divider until about 5 lb. remain. If a Jones divider is not available, the sample may be reduced by "quartering," but use of the divider is preferred. The sample shall be further reduced to approximately a size to pass a No. 10 sieve in laboratory rolls and again cut down by a Jones divider or by "quartering" to about 300 g. This sample shall then be divided in the same way into four portions of approximately 75 g. each and three of these shall be reserved. The fourth portion shall be pulverized until all has been passed through a No. 80

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

or No. 100 sieve. A steel mortar with a close-fitting pestle, both of hardened alloy steel, shall be used for this reduction. Rejection of any part of the sample because of failure to pass the screen is almost sure to produce error. The sample shall be thoroughly mixed, dried for one hour at 105 to 110° C. It is then ready for analysis. If necessary, one-half rather than one-fourth of the 10-mesh sample shall be pulverized to pass a No. 80 or No. 100 sieve and the resulting sample divided for distribution to the interested parties.

HIGH-CARBON FERRO-CHROMIUM, CHROMIUM METAL, LOW-CARBON FERRO-MANGANESE, MANGANESE METAL, FERRO-TUNGSTEN, FERRO-MOLYBDENUM AND FERRO-VANADIUM

These alloys are usually shipped in stout kegs or barrels. Most of these products are relatively costly, and accuracy in sampling is extremely important even when the tonnage involved is small.

If the shipment is believed to be fairly uniform in composition and contains only a small percentage of fines, a satisfactory sample can be obtained by removing the ends or cutting the sides of the packages and taking from each at least 0.5 per cent of the contents. Thus at least $2\frac{1}{2}$ lb. would be taken from a package containing 500 lb. If the product has been crushed to a small size, for example $\frac{1}{4}$ to $\frac{3}{4}$ in., the sample can be taken with a shovel or scoop. If the material is in larger pieces, as is usual with high-carbon ferro-chromium, the sample shall be taken by breaking fragments from a number of lumps until the requisite weight has been obtained as already described for the sampling of lumps of ferro-silicon. The gross sample shall be obtained by combining the samples taken from the individual packages and crushed and worked down in the manner described for the preceding group of alloys.

The relatively simple procedure of the preceding paragraph is justified only when experience has shown that the product being sampled contains no more than a small percentage of "fines" and is relatively uniform in composition. The "fines" usually differ somewhat in composition from the lumps and always work toward the bottom of the container while in transit. Products like tungsten powder or pulverized ferro-silicon (80 to 85 per cent Si) are also liable to segregate while in transit and should be sampled in a manner to avoid error due to the accumulation of a heavy constituent in the bottom of the container. The term "relatively uniform in composition" applies to a shipment or a sub-lot of a shipment in which the composition of any package is approximately the same as that of any other. Frequently a shipment comprises two or more lots, each uniform in composition within itself, but differing in this respect from the other lots. In these cases, each lot shall be sampled separately. If the product is rather variable in composition or if it contains a considerable proportion of fines, all of the packages comprising the shipment or lot shall be emptied and from each shall be taken a sample amounting to at least 0.5 per cent of the contents. The percentage of fine and lump alloy in each barrel shall be estimated and the sampling conducted accordingly. In sampling a small lot which varies widely in composition within itself, it is good practice to empty all of the packages, combine the entire contents and mix the lot thoroughly. The pile shall then be "quartered" repeatedly until a sample is obtained approximating 5 per cent of the weight of the entire lot. The gross sample obtained by combining samples from the individual barrels or by "quartering" the entire shipment shall be crushed and worked down in the manner described for the group containing ferrosilicon.

LOW-CARBON FERRO-CHROMIUM

Low-carbon ferro-chromium is ordinarily shipped in stout kegs. The alloy usually contains about 70 per cent of chromium together with maximum contents of carbon ranging from 0.1 to 2 per cent. The combination of hardness and toughness characteristic of this product, particularly of the lower carbon grades, makes it the most difficult by far of any of the ferro-alloys to sample properly. In view of the great importance of the accurate determination of the carbon content—especially of the alloy containing a maximum of 0.1 per cent of carbon—the utmost care must be taken to avoid contamination of the sample with fragments of steel from the tools. Fortunately low-carbon ferro-chromium is usually uniform in composition and free from "fines."

If the alloy is known to be reasonably uniform in composition, 10 lumps shall be drawn from each package in the shipment or sub-lot. This can be accomplished by removing the head or cutting the side of the keg. If the composition is variable, each package of the shipment or sub-lot shall be emptied and about 20 lumps taken at random. The number of lumps should be varied according to the net weight of the package and the average size of the lumps found in it. One fragment about $\frac{1}{8}$ in. in diameter shall be broken from each lump of the gross sample, using a heavy hammer and a plate of hardened alloy steel. As nearly as possible each fragment should offer a cross-section of the lump from which it was taken. The resulting sample shall be crushed to approximately a size to pass a No. 10 sieve by means of a mortar and pestle of hardened alloy steel. This material shall be cut down with a Jones divider or "quartered" until 30 or 40 g. remain. This quantity shall then be pulverized to pass a No. 40 sieve by means of the mortar and pestle. Especially in pulverizing the grades relatively low in carbon, an air hammer is almost indispensable. The material passing a No. 40 sieve shall be split into two or four parts according to the requirements, by means of a Jones divider or by "quartering." The following procedure is recommended for those cases in which prolonged crushing has resulted in a powder which passes entirely through a No. 20 sieve but which passes a No. 40 sieve in part only. The entire sample shall be passed through a No. 20 sieve and then divided into fractions finer and coarser than No. 40. The fractions shall be weighed and preserved in separate packages upon which are recorded the percentages by weight of the two sizes. The fractions may be analyzed separately or a composite sample may be prepared for the determination of each element, by weighing and subsequently combining the proper proportions of the coarse and fine material.

REQUIREMENTS FOR MORTAR AND PESTLE

In the analysis of ferro-alloys, errors arising from contamination of the sample by iron from the sampling appliances are both serious and common. A rapid and accurate method for the determination of unalloyed metallic iron in ferro-chromium and in ferro-silicon is known, and by this means such contamination has been detected and measured. Contamination has often been due to iron from the mortar and pestle but can be avoided by the proper selection and treatment of the steel from which they are made. These tools are essential to the accurate sampling of ferro-alloys—especially low carbon ferro-chromium—and when made in accordance with the following requirements are suitable for this purpose.

Chromium-nickel steel containing approximately 1.5 per cent Cr, 3 per cent Ni, and 0.35 per cent C is recommended for both tools. For the mortar a cylinder 3 by 3 in. shall be drilled axially to a diameter of $1\frac{1}{2}$ in. and a depth of $2\frac{1}{2}$ in. The bottom of the hole shall be made perfectly flat and the entire inner surface brought to a smooth finish. The pestle shall be made from a rod originally $1\frac{1}{2}$ in. in diameter by 5 in. long, and shall be smoothly faced and turned to a final diameter slightly less than $1\frac{1}{2}$ in. The mortar and pestle shall be heated for several hours in a laboratory muffle furnace at 750 to 800° C. and hardened by quenching in oil. The upper part of the pestle shall be slightly softer than the remainder in order to decrease the tendency to shatter. Both tools shall be polished with abrasive paper for the removal of scale. Such a mortar and pestle, together with an air hammer will pulverize alloys which will resist other equipment.

Other approved methods for the preparation of the pulverized sample from the gross sample may be substituted for the above provided they do not lead to contamination or loss of any portion of it. For example, the gross sample may be crushed entirely by rolls or in the absence of power machinery may be broken by hand to the required size. In the entire absence of power equipment the gross sample should be sent to a point where suitable facilities are available.

TENTATIVE METHODS OF CHEMICAL ANALYSIS OF FERRO-ALLOYS[1]

Serial Designation: A 104 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925

FERRO-SILICON

DETERMINATION OF SILICON BY FUSION WITH SODIUM PEROXIDE AND DEHYDRATION WITH HYDROCHLORIC ACID

Transfer 0.5000 g. (use 0.4000 g. for 90-per-cent silicon) of the finely powdered sample (passing a No. 150 sieve) to a 50-cc. pure iron crucible made from No. 20 gage (0.32 in. thickness) ingot iron, add approximately 13 g. of sodium peroxide. Mix *thoroughly* with a platinum or iron rod and carefully clean the rod of adhering particles by scraping with another rod. Cover the mixture with a layer of about 2 g. of additional peroxide. Heat the crucible and contents on a hot plate for 5 to 10 minutes to expel any water in the peroxide which would cause spattering in the subsequent fusion. Carefully fuse over a low flame by holding the crucible with a pair of tongs and slowly revolving it around the outer edge of the flame until the contents have melted down quietly. When the fusion is molten rotate the crucible carefully to stir up any unattacked particles on the bottom or sides, the crucible and contents being maintained at a low red heat. Just before completion of the fusion, which requires only three or four minutes, increase the temperature to bright redness for a minute. If the reaction has proceeded too violently, due to too rapid heating, to the use of insufficient sodium peroxide, or to the lack of thorough mixing, appreciable loss will occur and the work should be repeated.

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

Cover the crucible and allow it to cool until the fusion has solidified, and tap the crucible, while still warm, several times on an iron plate, which will loosen the fused mass in a solid cake. When the crucible and cake have completely cooled, transfer the cake to a large (275-cc.) gold or preferably platinum dish. Cover the dish with a clock glass and cautiously add 50 cc. of cold water. When the reaction ceases, wash the small amount of adhering matter in the crucible into the dish with a little water. Cool the solution and carefully add an excess of hydrochloric acid. Evaporate to dryness, preferably on a steam bath, but do not bake at a temperature above 110° C. If platinum or gold dishes are not available for solution of the fused cake, the latter can be disintegrated with water in a pure nickel dish and the contents then transferred to a porcelain dish containing sufficient hydrochloric acid to provide an excess of acid. It is not desirable to dissolve the fusions directly in porcelain, because of the action of the alkaline solution.

When the residue is dry, allow the dish to cool. Add 10 cc. of hydrochloric acid (sp. gr. 1.13), cover the dish and heat gently for a few minutes. Dilute with 100 cc. of hot water, digest for a few minutes and filter through an 11-cm. paper. Wash about eight times with hot 5-per-cent hydrochloric acid, three times with warm 5-per-cent sulfuric acid and a few times with hot water. Reserve this paper until the second evaporation has been completed.

Evaporate the filtrate to dryness and bake the covered casserole or dish at 110° C. for one hour. Heating at a higher temperature is unnecessary and also disadvantageous. Cool, add 10 cc. of hydrochloric acid (1:1), digest on the steam bath for ten minutes, add 100 cc. of warm water, filter immediately and wash about eight times with cold 1-per-cent hydrochloric acid, twice with cold 5-per-cent sulfuric acid and a few times with cold water.

Place the paper and silica from the *second* dehydration in a large partially covered platinum crucible and char the paper without inflaming. Add the paper containing the silica from the first dehydration, char as before and then ignite carefully until the carbon has been burned off. Great care should be exercised in igniting the papers as the current of air produced by a burning filter is sufficient to carry finely divided silica out of the crucible. When the carbon of the filter paper has been completely burned, gradually heat the tightly covered crucible to the full heat of a blast lamp and continue the heating at 1050 to 1100° C. for 25 minutes. Cool in a desiccator, weigh and check for constant weight by ten minutes' additional heating at the above temperature. Add sufficient sulfuric acid (1:1) to moisten the silica and then 15 to 20 cc. of pure hydrofluoric acid (48-per-cent). Evaporate to dryness, ignite and weigh. The loss represents silica which contains 46.72 per cent of silicon. A blank, not omitting the same amount of hydrofluoric acid, should be carried through all stages of the operation.

NOTE

The silica remaining in the filtrate after the second dehydration is practically negligible, averaging about 0.0006 to 0.0010 g. in hydrochloric dehydration and 0.0010 to 0.0015 g. in sulfuric dehydration.

FERRO-MANGANESE

DETERMINATION OF MANGANESE
BY THE
MODIFIED BISMUTHATE METHOD[1]

REAGENTS REQUIRED

Dilute Nitric Acid (sp. gr. 1.135).—Mix 300 cc. of nitric acid (sp. gr. 1.42) and 700 cc. of distilled water. Test for proper specific gravity.

Dilute Nitric Acid (3 : 97).—Mix 30 cc. of recently boiled nitric acid (sp. gr. 1.42) and 970 cc. of distilled water. Add a little sodium bismuthate and shake well.

Asbestos.—Asbestos for this work should be digested in hot nitric acid (sp. gr. 1.42) and then washed free from acid with distilled water.

Sodium Bismuthate.—This reagent should be of 80-per-cent grade and its oxidizing power tested as follows: Shake 0.5 g. of the reagent with 4 g. of potassium iodide and a little water in a stoppered flask. Add 15 cc. of hydrochloric acid and allow to stand in the dark, with occasional shaking until the bismuthate has entirely decomposed. Dilute to 300 cc. and titrate with 0.1 *N* sodium thiosulfate using starch as an indicator at the end. One cubic centimeter of 0.1 *N* $Na_2S_2O_3$ equals 0.0140 g. of $NaBiO_3$.

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[1] *Journal of Industrial and Engineering Chemistry*, Vol. 16, p. 58 (1924).

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Standard Potassium Permanganate Solution.—See the Determination of Chromium in Ferro-Chromium.[1] One cubic centimeter of 0.1 *N* $KMnO_4$ equals 0.001099 g. of manganese.

METHOD

(*A*) *Solution of the Sample*

All samples should be crushed to approximately a size to pass a No. 100 sieve and dried at 105 to 110° C.

With high-carbon 80-per-cent ferro-manganese, dissolve 0.25 g. of the prepared sample in 60 cc. of nitric acid (sp. gr. 1.42) in a 750-cc. Erlenmeyer flask, provided with a clock-glass cover or trap to prevent loss by spraying.

Add 2 g. of potassium nitrate and boil gently until the volume of the solution has been reduced to about 15 cc. Cool, add 10 cc. of H_2SO_4 (1:1) and evaporate to strong fumes of sulfuric acid. Cool somewhat, dilute the solution with 200 cc. of HNO_3 (sp. gr. 1.135), heat to boiling, remove from the heat and add sodium bismuthate a little at a time until a precipitate of manganese dioxide has formed. When this occurs, add sulfurous acid dropwise until the precipitate has dissolved and an excess of about 1 cc. has been provided to reduce any chromium to the trivalent form. Boil the solution about five minutes to completely expel oxides of nitrogen, dilute to 250 cc. with water, cool the flask and solution in crushed ice and treat as described later. High-carbon ferro-manganese containing 1 per cent or more of chromium may not dissolve completely in nitric acid. In this case, dilute the solution after treatment with nitric acid, filter, and fuse the washed residue with a little sodium carbonate. Dissolve the melt in a small volume of nitric acid (sp. gr. 1.135), and add it to the main solution. Treat with potassium nitrate, etc., as previously described.

With low-carbon ferro-manganese or manganese metal dissolve 0.2500 g. of the sample in 250 cc. of nitric acid (sp. gr. 1.135) in a covered 750-cc. Erlenmeyer flask and boil the solution gently for a few minutes to expel oxides of nitrogen. Treat with bismuthate and sulfurous acid as described above.

With silico-manganese (60 to 70 per cent of manganese) and manganese-silicon (20 to 25 per cent of manganese) treat 0.3000 g. of the former and 1.000 g. of the latter with 5 cc. of hydrofluoric acid in a large platinum dish equipped with a gold or platinum cover. (Some spiegeleisens are completely soluble in nitric acid in which case the sample can be dissolved directly in 250 cc. of nitric acid (sp. gr. 1.135). When the reaction moderates, add nitric acid (sp. gr. 1.42) a few drops at a time until the sample has completely dissolved. Add 10 cc. of nitric acid (sp. gr. 1.42) and evaporate to a syrupy consistency. Again add 10 cc. of nitric acid (sp. gr. 1.42) and evaporate as before to expel hydrofluoric acid. Dissolve the residue in nitric acid (sp. gr. 1.135), transfer to a 750-cc. Erlenmeyer flask, dilute with nitric acid (sp. gr. 1.135) to 250 cc. and treat as described before with sodium bismuthate, etc.

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[1] See p. 550.

(*B*) *Oxidation of Manganese*

If the preceding directions have been followed, the manganese will be present in a concentration of approximately 0.001 g. per 1 cc. of nitric acid (sp. gr. 1.135). This concentration of manganese and nitric acid, together with a temperature of 10 to 15° C. are the conditions which insure maximum stability of the permanganic acid, formed as further described. To insure complete oxidation of the manganese to permanganic acid, it is essential that the sodium bismuthate (80 per cent of $NaBiO_3$) be used in the ratio of at least 26 g. to every gram of manganese in solution. Add approximately 7.0 g. of sodium bismuthate (80-per-cent) to the flask, *agitate briskly* for one minute, dilute with 250 cc. of cold water, and filter immediately on a layer of acid-washed asbestos supported on a 2-in. alundum or perforated porcelain plate resting in a large glass funnel. The filter can be washed free from manganese more readily if not allowed to run dry during the filtering and washing. Wash the filter and residue with cold 3-per-cent nitric acid until the washings are entirely colorless and immediately treat the filtrate and washings as described in the following paragraph.

Weigh accurately 9.000 g. of ferrous ammonium sulfate, taken from a bottle of the well-mixed salt, and add to the filtered solution of permanganic acid. Stir briskly and as soon as the reduction is complete and the salt has dissolved, titrate the excess of ferrous salt with 0.1 *N* potassium permanganate. The manganese value of the ferrous salt is obtained in the following manner:

Two hundred and fifty cubic centimeters of nitric acid (sp. gr. 1.135) contained in a 750-cc. Erlenmeyer flask are treated with approximately 2.0 g. of sodium bismuthate and the solution is vigorously agitated for one minute, diluted with 250 cc. of cold water, and immediately filtered on a layer of acid-washed asbestos as previously described. Five (5.000) grams of the ferrous ammonium sulfate are added to the filtrate and the resulting solution is immediately titrated with 0.1 *N* $KMnO_4$ until a faint pink color appears. The excess of permanganate used to cause an end point color must be determined by watching the color in another beaker containing the same amounts of acid and cold water.

NOTES

1. Chromium when less than 2 per cent does not cause any interference if the reactions are rapidly done in cold solutions. Larger amounts interfere to some extent and should be separated prior to the final oxidation with bismuthate.

2. The gravimetric determination of manganese by the pyrophosphate method will give high values if the precipitate of manganese ammonium phosphate is allowed to stand more than ten hours before filtration.

3. Volumetric methods such as Volhard's, Ford-Williams' and Julian's do not permit the use of a theoretical titre. Each necessitates the use of an empirical titre obtained by titration of material of known manganese content which has been carried through all steps of the method.

DETERMINATION OF CARBON BY THE DIRECT COMBUSTION METHOD

See the Determination of Carbon in Ferro-Chromium by the Direct Combustion Method.[1] A furnace temperature of 1050 to 1075° C. suffices, and high-silicon material should be covered with 1 or 2 g. of ingot iron.

DETERMINATION OF PHOSPHORUS BY THE ALKALIMETRIC METHOD

SOLUTIONS REQUIRED

Dilute Nitric Acid (sp. gr. 1.135).—Mix 300 cc. of HNO_3 (sp. gr. 1.42) and 700 cc. of distilled water.

Ammonium Molybdate.—See the Determination of Phosphorus in Ferro-Vanadium by the Modified Molybdate-Magnesia Method.[2]

Standard Sodium Hydroxide.—Dissolve 6.5 g. of purified NaOH in 1000 cc. of distilled water, add a slight excess of a 1-per-cent solution of $Ba(OH)_2$, let stand for 24 hours, decant the liquid and standardize against Bureau of Standards Standard Benzoic Acid as follows: Dissolve 0.6 g. of benzoic acid in 15 cc. of alcohol in a small Erlenmeyer flask and titrate in the cold with phenolphthalein indicator, taking precautions to prevent access of carbon dioxide from the air during titration. The ratio of phosphorus to sodium hydroxide is taken as 1 to 23 in calculating the value of the sodium hydroxide. Protect the solution from carbon dioxide with a soda-lime tube.

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[1] See p. 553.

[2] See p. 556.

Standard Nitric Acid.—Mix 10 cc. of HNO_3 (sp. gr. 1.42) and 1000 cc. of distilled water. Titrate the solution against the standard hydroxide solution, using phenolphthalein as an indicator. Adjust the solution so that it is equivalent to the sodium hydroxide solution.

Dilute Ammonium Hydroxide (sp. gr. 0.96).—Dilute 400 cc. of NH_4OH (sp. gr. 0.90) with 600 cc. of water. The strength of this solution should be established by titration or test with a hydrometer.

METHOD

Dissolve 2.000 g. of the sample in 70 cc. of nitric acid (sp. gr. 1.42), evaporate to dryness and bake for two or three hours at 120° C. High-silicon alloys may require the addition of a little hydrofluoric acid for complete decomposition. In this case, several evaporations with 15-cc. portions of nitric acid (sp. gr. 1.42) should be made to remove hydrofluoric acid before the baking treatment. Moisten the residue with 25 cc. of dilute hydrochloric acid (1:1), digest a few minutes,[1] add 75 cc. of water, heat until the salts are dissolved and then filter to remove silica. Evaporate the filtrate to a syrupy condition, add 15 cc. of nitric acid (sp. gr. 1.42) and again evaporate to a small volume. Again add 15 cc. of nitric acid (sp. gr. 1.42) and evaporate to a small volume. Add 100 cc. of nitric acid (sp.gr. 1.135), heat to boiling and add a 1.5-per-cent solution of potassium permanganate until oxides of manganese are precipitated. Dissolve the oxides of manganese by addition of sulfurous acid and boil to expel the oxides of nitrogen. To the cold solution add 40 cc. of NH_4OH (sp. gr. 0.96) and 50 cc. of ammonium molybdate solution and shake for 5 minutes. Allow to settle for 10 minutes; for material containing less than 0.02 per cent of phosphorus, allow to stand 30 minutes. Filter and wash 10 times with a 1-per-cent solution of KNO_3. Return the precipitate and paper to the precipitating vessel, add 1 to 3 cc. in excess of standard NaOH solution and 25 cc. of water, both free from carbon dioxide, and shake or stir until the precipitate is dissolved. Dilute to 100 cc. with water free from carbon dioxide, add 3 drops of a 0.2-per-cent solution of phenolphthalein, and discharge the pink color with standard acid. Finish the titration by adding standard alkali until the reappearance of the pink color. With very low phosphorus material this titration should be corrected by a blank carried through all steps of the method.

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[1] Addition of a little sulfite or sulfurous acid facilitates solution after the baking treatment.

NOTE

If vanadium is present in amounts over 0.08 per cent it should be reduced by ferrous sulfate and sulfurous acid before precipitation with molybdate.

DETERMINATION OF SULFUR
BY THE
OXIDATION METHOD

Proceed as in the Determination of Sulfur by the Oxidation Method as described in the Standard Methods of Chemical Analysis of Plain Carbon Steel (Serial Designation: A 33) of the American Society for Testing Materials.[1]

NOTES

Routine determinations of sulfur can be made by the evolution method provided concentrated hydrochloric acid (sp. gr. 1.19) is used, preferably in all-glass apparatus of the Pulsifer type.

DETERMINATION OF SILICON
BY THE
NITRO-SULFURIC ACID METHOD

SOLUTIONS REQUIRED

Nitro-Sulfuric Acid.—Pour 160 cc. of sulfuric acid (sp. gr. 1.84) slowly and with stirring into 660 cc. of water and add 180 cc. of nitric acid (sp. gr. 1.42).

METHOD

Dissolve 0.9344 g. of the sample passing a No. 100 sieve with 50 cc. of the nitro-sulfuric acid in a 250-cc. porcelain casserole and evaporate until copious fumes of sulfuric acid are evolved. After the solution has cooled somewhat add 125 cc. of distilled water and 5 cc. of HCl (sp. gr. 1.19). Heat with frequent stirring until all salts are dissolved and immediately filter on a 9-cm. paper. Wash the precipitate with cold dilute HCl (5:95) and hot water alternately to complete the removal of soluble salts, and finally with hot water until free from acid. Transfer the filter to a platinum crucible, char the paper carefully, and finally ignite with the crucible covered over a blast lamp or in a muffle furnace at 1050 to 1100° C. for at least 10 minutes; cool in a desiccator and weigh. Add sufficient dilute sulfuric acid (1:1) to moisten the silica and then a small amount of hydrofluoric acid. Evaporate to dryness, ignite and weigh. The difference in weights divided by 2 and multiplied by 100 represents the percentage of silicon in the alloy.

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[1] 1924 Book of A.S.T.M. Standards.

NOTE

For silico-manganese and manganese-silicon, fuse 0.4672 g. of the former or 0.2336 g. of the latter with 8 g. of sodium peroxide in a pure iron crucible. Dissolve the fusion and evaporate to fumes of sulfuric acid. When acidifying the solution of the fusion with sulfuric acid it is necessary to add some sulfurous acid to dissolve the manganese dioxide which separates out.

FERRO-CHROMIUM

DETERMINATION OF CHROMIUM
BY
FUSION WITH SODIUM PEROXIDE

SOLUTIONS REQUIRED

Potassium Permanganate, 0.1 N.—Dissolve 3.2 g. of potassium permanganate in 1000 cc. of distilled water, allow to age for at least ten days and filter through purified asbestos. Standardize the permanganate solution against Bureau of Standards Standard Sodium Oxalate as follows:

In a 400-cc. beaker dissolve 0.25 to 0.30 g. of sodium oxalate in 200 to 250 cc. of hot water (80 to 90° C.) and add 10 cc. of dilute H_2SO_4 (1:1). Titrate at once with the permanganate solution, stirring the liquid vigorously and continuously. The permanganate must not be added more rapidly than 10 to 15 cc. per minute, and the last 0.5 to 1 cc. must be added dropwise, with particular care to allow each drop to fully decolorize before the next is introduced. The excess of permanganate used to cause an end point color must be estimated by matching the color in another beaker containing the same bulk of acid and hot water. The temperature of the solution should not be below 60° C. by the time the end point is reached. One cubic centimeter of 0.1 *N* $KMnO_4$ is equivalent to 0.001734 g. of chromium.

Standard Ferrous Ammonium Sulfate.—Dissolve 43 g. of the salt in 250 cc. of cold water, add 40 cc. of sulfuric acid (1:3), dilute to one liter with distilled water, and mix thoroughly. Large amounts of the solution can be conveniently mixed by means of a stream of carbon dioxide from a cylinder. As the ferrous ammonium sulfate solution gradually weakens in reducing power it is necessary to standardize it daily or at the same time the ferro-chromium is analyzed.

The solution can be standardized against either potassium dichromate or sodium oxalate.

The first method is somewhat simpler and more satisfactory for routine work as the conditions prevailing in both the standardization and in the analysis are practically identical. No "blank" corrections are afterwards necessary where the amounts of dichromate, ferrous salt and permanganate are approximately those prevailing in the analysis. The second method is employed chiefly for checking the accuracy of the results obtained with potassium dichromate and in cases where only occasional analyses are made and pure dichromate is not readily available.

To prepare pure potassium dichromate, recrystallize at least twice the purest grade of the salt obtainable, dry the crystals at 150° C., grind to a fine powder and again dry at 150° C. to constant weight. Keep the prepared salt in a ground-glass stoppered bottle. Transfer 1.0000 g. of the pure dichromate, which contains approximately the same amount of chromium as an 0.5000-g. sample of 70-per cent ferro-chromium, to an 800-cc. beaker. Dissolve in cold water, acidify with 40 cc. of sulfuric acid (1:3) and dilute with cold water to 500 cc. Add a slight excess of the ferrous sulfate solution, approximately 190 cc., and titrate the excess ferrous salt with 0.1 N permanganate to the first faint permanent darkening of the clear green color.

Example.—The calculations involved are illustrated as follows: In a standardization, 190.0 cc. of the ferrous solution was used and 5.06 cc. of 0.1 N permanganate was required to titrate the excess.

1 cc. of 0.1 N $K_2Cr_2O_7$ contains 0.004903 g. of $K_2Cr_2O_7$

1 g. of $K_2Cr_2O_7$ equals 203.94 cc. of 0.1 N $K_2Cr_2O_7$

then

$203.94 + 5.06 = 209.0$ cc. of equivalent 0.1 N solution reduced by the ferrous solution.

$$\frac{209.0}{190} = 1.100, \text{ the 0.1 } N \text{ normality factor.}$$

In standardizing through sodium oxalate, it is necessary to add the ferrous ammonium sulfate solution by means of an accurately calibrated pipette or burette instead of an automatic pipette. Transfer 100 cc. of the approximately 0.1 N ferrous solution to a 600-cc. beaker, dilute to 300 cc. with cold 5-per-cent sulfuric acid, add 2 cc. of phosphoric acid and titrate immediately with 0.1 N $KMnO_4$ to a faint permanent pink color. Determine the "blank" on the same volume of water and acids and deduct. When this method is used it is necessary in the actual analysis to determine a "blank" to counteract the influence of the green color of the chromium sulfate upon the permanganate end point. This "blank" can be determined in the solution used in the analysis and is obtained as described later.

METHOD

Transfer 0.5000 g. of the sample of ferro-chromium (dried at 105 to 110° C.) to a 30-cc. heavy-walled porcelain crucible. Samples of low-carbon ferro-chromium should be crushed to pass through a No. 40 screen, while high-carbon ferro-chromium and chromium metal should be reduced to pass a No. 100 screen. Add approximately 8 g. of sodium peroxide and thoroughly mix the contents of the crucible with a small rod, being careful to clean the rod, which can be done conveniently by scraping with another rod. Cover the mixture with 1 to 2 g. of sodium peroxide. Carefully fuse the contents of the covered crucible, preferably in an electric muffle heated to 600 to 700° C. Fusion for five minutes at a low red heat after the mass has melted will insure complete decomposition. When the charge has melted, the crucible should be given a slight rotary motion to stir up any unattached particles.

Place the cooled crucible and cover in a 600-cc. beaker and dissolve the contents in 200 cc. of water. When the melt has dissolved, remove the crucible and cover, add 1 g. of sodium peroxide and boil the solution ten minutes to completely decompose the excess of peroxide.

Allow the precipitate to settle and filter through a pad of ignited asbestos, preferably in a Büchner funnel. An asbestos rather than a paper filter should be used, for the alkaline solution extracts reducing matter from paper which would cause low results for chromium. Thoroughly wash the precipitate with hot water, and at no time allow the precipitate to run dry during the filtering and washing. If desired, the precipitate can be subsequently dissolved in dilute sulfuric acid and tested for chromium by adding a slight excess of sodium peroxide; the amount of chromium retained is negligible if the precipitate is filtered and washed as directed.

Cool the filtrate, acidify with 50 cc. of sulfuric acid (1:1), again cool to room temperature, transfer to an 800-cc. beaker, dilute with cold water to 500 cc. and add a measured excess of ferrous ammonium sulfate. The point at which reduction is complete can be told by changes in the deep green color which is developed. The ferrous salt can be added either as an approximately 0.12 *N* solution or in weighed portions from a bottle of the well-mixed salt. If the solution of ferrous salt is used, approximately 180 cc. will be required for a 70-per-cent alloy; if the salt is added, 8 g. will usually be sufficient. Titrate the excess of ferrous salt with standard 0.10 *N* potassium permanganate. The first faint permanent darkening of the clear green color is taken as the end point, which is quite sharp and well defined to the practiced eye. If the ferrous salt has been standardized against dichromate, subtract the required number of cubic centimeters of 0.1 *N* permanganate from the number of cubic centimeters of 0.1 *N* ferrous solution (obtained by multiplying the number of cubic centimeters of the approximately 0.1 *N* ferrous solution by the 0.1 *N* normality factor). The difference represents the number of cubic centimeters of 0.1 *N* ferrous solution which multiplied by 0.001733 g. gives the number of grams of chromium present.

Where the ferrous solution has been standardized against sodium oxalate, it is necessary to subtract a "blank" from the permanganate titration. This "blank" is obtained as follows: When the end point is reached and the readings have been noted, add about 5 cc. in excess of the ferrous solution and oxidize the excess of ferrous iron by adding 8 cc. of a 15-per-cent solution of ammonium persulfate. *Stir the liquid vigorously for one minute.* Carefully add 0.1 *N* permanganate to the solution with constant stirring until the same end point is obtained as before. The volume of permanganate required, usually 0.2 to 0.4 cc., constitutes the "blank" to be deducted from the volume of the permanganate titration. For example, in the determination of chromium in an 0.5000-g. sample, 190.0 cc. of a 0.11 *N* ferrous solution and 11.00 cc. of an 0.1 *N* permanganate (11.40 – 0.4 cc. blank) were employed.

The calculations are as follows:

190.0 × 1.100 = 209.0 cc. of 0.1 *N* ferrous solution
209.0 – 11.0 = 198.00 cc.
198.0 × 0.001734 × 2 × 100 = 68.65 per cent of chromium

The above calculations are given for exactly 0.1 *N* permanganate; if the permanganate is not 0.1 *N*, the volume used must be converted to the equivalent of 0.1 *N* by multiplying by the proper factor.

DETERMINATION OF CARBON BY THE DIRECT COMBUSTION METHOD

Determine carbon by the direct combustion method described in the Standard Methods of Chemical Analysis of Plain Carbon Steel (Serial Designation: A 33), with the following modifications:

1. Ferro-chromium alloys require a furnace temperature of 1125 to 1150° C. in order to yield all of the carbon in the material.

2. The determination is made as given in the reference except that the weighed sample of ferro-chromium, 0.5000 g. for 4 to 8-per-cent carbon and 3 to 1 g. for 0.10 to 2.00-per-cent carbon, is mixed

with 1 g. of copper oxide and covered with 2 g. of pure ingot iron drillings in order to facilitate complete combustion.

3. On account of the small weight of sample used, the blanks are important and must be carefully determined.

DETERMINATION OF SILICON

Low-carbon ferro-chromium and chromium metal can be decomposed by means of dilute sulfuric acid, but the high-carbon alloy has to be fused with sodium peroxide. For low-carbon ferro-chromium, transfer 0.9344 g. (a double factor weight) to a 250-cc. porcelain casserole, add 60 cc. of sulfuric acid (1:4) and warm the covered casserole gently until the reaction is complete. For high-carbon ferro-chromium, transfer 0.9344 g. of the sample passing a No. 100 sieve to a 30-cc. pure iron crucible made from No. 20 gage (0.032 in. thickness) ingot iron. Add approximately 8 g. of sodium peroxide, mix thoroughly and fuse as directed under the determination of chromium. When the fusion has solidified, tap the covered crucible, while still warm, on an iron plate to loosen its contents in a solid cake which is then transferred to a 250-cc. casserole. Rinse the crucible with a little water, transfer the solution to the casserole, cover, and carefully add 60 cc. of sulfuric acid (1:4) after which the cover glass is rinsed and removed.

Evaporate the solution obtained by either of the above procedures until fumes of sulfuric acid are given off. If chromium sulfate solutions are fumed at too high temperatures or for any great length of time or with too little free acid present, considerable difficulty will occur in dissolving the salts. The fuming should, therefore, be made at a temperature not much higher than that required for evolving the acid. Allow the casserole and contents to partly cool (if permitted to become entirely cold, difficulty may be experienced in getting chromium salts to dissolve), add 100 cc. of warm water while stirring to prevent the residue from caking on the bottom and boil the solution gently for several minutes to dissolve all salts. Filter, washing the paper several times with cold dilute hydrochloric acid (2-per-cent), and then with warm water. Transfer the filter and contents to a platinum crucible, char the paper, carefully ignite to remove carbon, and then heat the covered crucible over a blast lamp or in a muffle furnace at 1050 to 1100° C. for at least 15 minutes. Cool in a desiccator and weigh. Add sufficient dilute sulfuric acid (1:1) to moisten the silica and then a small amount of hydrofluoric acid. Evaporate to dryness, ignite and weigh. The difference in weights represents silica which contains 46.72 per cent of silicon.

NOTE

Crucibles made from pure ingot iron contain only traces of silicon and the amount of silica present in the sodium peroxide used is usually negligible, but "blanks" should be run on new lots of crucibles and of peroxide. All nickel crucibles, even the German "Rein" nickel, contains appreciable amounts of silicon.

FERRO-VANADIUM

DETERMINATION OF VANADIUM
BY THE
AMMONIUM PERSULFATE METHOD

SOLUTIONS REQUIRED

Ammonium Persulfate (15 per cent).—Make up as needed by dissolving 15 g. of the salt in 100 cc. of distilled water. This salt should be 95-per-cent pure and its purity tested by adding 30 cc. of a standard ferrous sulfate solution to 0.3 g. of the persulfate, then 200 cc. of hot water and titrating the excess ferrous salt with potassium permanganate. One cubic centimeter of 0.1 N ferrous solution equals 0.01141 g. of $(NH_4)_2S_2O_8$.

Potassium Ferricyanide Indicator.—Dissolve 0.1 g. of the salt in 100 cc. of water. This solution should be made up fresh daily.

Standard Ferrous Ammonium Sulfate, approximately 0.1 N.—See the Determination of Chromium in Ferro-Chromium.[1]

Potassium Permanganate, 0.1 N.—See the Determination of Chromium in Ferro-Chromium.[1] One cubic centimeter of 0.1 N $KMnO_4$ is equivalent to 0.005096 g. of vanadium in the following method.

METHOD

Treat 0.5000 g. of the alloy, dried at 105 to 110° C., in a 250-cc. covered beaker with a mixture of 60 cc. of sulfuric acid (1:3) and 25 cc. of nitric acid (1:1). If the alloy fails to decompose readily because the silicon content is high, it may be necessary to add a small amount of hydrofluoric acid. In this event, a 200-cc. platinum dish should be substituted for the beaker. When the reaction has abated, rinse the cover glass and evaporate the solution until copious fumes of sulfuric acid are evolved. Cool, add 100 cc. of water and heat until the salts are dissolved. Transfer the solution to an 800-cc. beaker, dilute to 400 cc. and to the cooled solution add an approximately 0.1 N solution of potassium permanganate until a strong pink color has developed which remains permanent for thirty seconds.

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[1] See p. 550.

Next reduce the vanadium by the use of an approximately 0.1 *N* solution of ferrous ammonium sulfate added until a drop of the solution withdrawn from the beaker and placed on a drop of potassium ferricyanide indicator shows ferrous iron present—about 35 cc. of 0.1 *N* ferrous solution. Add 5 cc. of the ferrous ammonium sulfate in excess, stir the solution at least one minute, oxidize the excess of ferrous sulfate with 8 cc. of 15-per-cent ammonium persulfate and stir the solution vigorously for *one minute*. Titrate the solution with 0.1 *N* potassium permanganate solution, added with constant stirring until a faint pink color appears which remains permanent for 30 seconds. Determine the "blank" by dissolving 0.4 g. of "ingot iron" in 60 cc. of H_2SO_4 (1:3) and 5 cc. of nitric acid (sp. gr. 1.42), and putting the solution through all of the operations of the analysis. If the alloy contains an appreciable amount of chromium, a weight of pure $K_2Cr_2O_7$ equivalent to the amount of chromium in the sample is dissolved in the acids with the ingot iron.

DETERMINATION OF CARBON

See the Determination of Carbon in Ferro-Chromium by the Direct Combustion Method.[1] A furnace temperature of 1050 to 1100° C. will suffice for ferro-vanadium and complete combustion except in very high-silicon material can be secured without the addition of ingot iron and copper oxide.

DETERMINATION OF PHOSPHORUS
BY THE
MODIFIED MOLYBDATE-MAGNESIA METHOD

SOLUTIONS REQUIRED

Ammonium Molybdate.—

Solution No. 1.—Place in a beaker 100 g. of 85-per-cent molybdic acid, mix thoroughly with 240 cc. of distilled water, add 140 cc. of NH_4OH (sp. gr. 0.90), filter, and add 60 cc. of HNO_3 (sp. gr. 1.42).

Solution No. 2.—Mix 400 cc. of HNO_3 (sp. gr. 1.42) and 960 cc. of distilled water.

When the solutions are cold, add Solution No. 1 to Solution No. 2, stirring constantly, then add 0.1 g. of ammonium phosphate dissolved in 10 cc. of distilled water, and let stand at least 24 hours before using.

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[1] See p. 553.

Magnesia Mixture.—Dissolve 50 g. of $MgCl_2 \cdot 6H_2O$ and 200 g. of NH_4Cl in 1000 cc. of distilled water containing a few cubic centimeters of hydrochloric acid.

METHOD

Transfer 4 g. of the sample to a platinum dish of 150 to 200-cc. capacity, add 70 cc. of dilute HNO_3 (5:6) and heat the covered dish until vigorous action ceases. Add 1 or 2 cc. of hydrofluoric acid, stir the solution, evaporate to dryness, moisten with 10 cc. of nitric acid (sp. gr. 1.42) and again evaporate to dryness. Moisten the cooled residue with 10 cc. of HNO_3 (sp. gr. 1.42), add 50 cc. of water and wash the contents of the dish into a 400-cc. beaker, rubbing the dish well with a policeman. Remove the last traces of adhering salts with 5 cc. of HCl (sp. gr. 1.19) and hot water. Add 10 cc. of HNO_3 (sp. gr. 1.42) and evaporate to a syrupy consistency. Cool, add 25 cc. of HNO_3 (sp. gr. 1.42) 60 cc. of water, 1 g. of boric acid (to take care of any residual fluoride) and heat gently until solution occurs. Filter through a rapid paper into a 300-cc. Erlenmeyer flask and wash with hot water. Heat the solution to boiling, add about 12 cc. of potassium permanganate solution (1.5-per-cent) and continue the boiling until manganese dioxide precipitates. Dissolve the precipitate by the addition of sufficient sulfurous acid added dropwise. About 1 cc. usually suffices. Boil for a few minutes to completely expel oxides of nitrogen. Add to the cold solution 15 cc. of ammonium hydroxide (sp. gr. 0.90) and cool the solution to 15° C.

Next reduce the vanadium in the cold (15° C.) solution by ferrous sulfate added in approximately 1.5 g. excess. The use of a 50-per-cent solution of the salt furnishes a convenient way of adding the necessary amount. One gram of quinquevalent vanadium requires 5.5 g. of $FeSO_4 \cdot 7H_2O$ for reduction to the quadrivalent state. A 4-g. sample of 30-per-cent vanadium alloy, including an excess of 1.5 g., would require 8.1 g. of ferrous sulfate. Too large an excess of ferrous sulfate should be avoided. The addition of ferrous sulfate will change the color of the solution to a deep blue. Add 2 drops of a saturated solution of sulfurous acid, 100 cc. of molybdate reagent, shake for 10 minutes and allow to stand at 15° C. for 4 to 6 hours, or preferably overnight. It is well to let the solution stand in a refrigerator. Filter on a paper of close texture, keeping as much of the precipitate in the flask as possible, and wash the flask and the precipitate with not more than 50 cc. of cold, dilute nitric acid (1:99). Set the filtrate and washings aside after thorough mixing and see whether further separation of phosphomolybdate occurs. Dissolve the precipitate on the filter in 20 cc. of dilute ammonium hydroxide (1:1) to which have been added 2 g. of citric acid, catch the solution in the original flask containing the bulk of the yellow precipitate and finally wash the filter several times with dilute hydrochloric acid (5:95). If the ammoniacal solution of the phosphomolybdate is not clear at this point, heat it to boiling, filter through the same paper, and wash the paper with hot water. In this case, and whenever appreciable amounts of such elements as titanium or zirconium are known to be present in the original material, preserve the paper and ignite it in a small platinum crucible with the paper reserved below.

Render the ammoniacal solution acid with hydrochloric acid, add 20 cc. of magnesia mixture and then ammonium hydroxide (sp. gr. 0.90) slowly until a crystallin precipitate appears, and then finally an excess of 3 to 5 per cent by volume. The volume of the solution at this point should not exceed 100 cc. Allow the solution to stand in a cool place for 4 to 6 hours, or preferably overnight.

Filter, keeping as much of the precipitate as possible in the flask, and wash the flask and the precipitate moderately with dilute ammonium hydroxide (1:20). Set the filtrate and washings aside after thorough mixing and see whether further precipitation occurs. Dissolve the precipitate on the filter in 25 cc. of dilute hydrochloric acid (1:1), catching the solution in the original flask containing the bulk of the precipitate. Wash the filter thoroughly with dilute hydrochloric acid (5:95). In case the original material contains appreciable amounts of the elements mentioned above (titanium or zirconium), add the paper to the one reserved above, ignite, fuse any residue with as little carbonate as possible (not over 0.5 g.), extract the cooled melt with hot water, filter, and add the water solution to the hydrochloric acid solution. Transfer the solution to a 200-cc. beaker. In case the amount of arsenic is known to be very small, add 0.5 to 1 g. of ammonium bromide and boil the hydrochloric acid solution to a volume of 5 to 10 cc. Where large amounts of arsenic were originally present, treat with hydrogen sulfide, digest, filter, and wash with acidulated hydrogen sulfide water. Boil to expel hydrogen sulfide and to reduce the volume to 50 to 75 cc. Add 0.1 to 0.2 g. of citric acid, 2 to 3 cc. of magnesia mixture, make ammoniacal as above, and allow to stand in a cool place for 4 to 24 hours.

Filter, wash with dilute ammonium hydroxide (1:20), and ignite the precipitate carefully and at as low a temperature as possible until the carbon has been destroyed and the residue is white. Finally, ignite at approximately 1000° C. to constant weight. Dissolve the ignited residue in 5 cc. of dilute nitric acid (1:1) and 20 cc. of water.

Filter, wash the residue with hot water, ignite and weigh. Treat with a few drops of hydrofluoric acid, evaporate to dryness, ignite and re-weigh. Subtract any loss of weight from the original weight of the pyrophosphate, and calculate on the basis of this purified magnesium pyrophosphate.

DETERMINATION OF SULFUR
BY THE
OXIDATION METHOD

Transfer 4.57 g. of the sample to a 600-cc. beaker and dissolve it in 75 cc. of HNO_3 (sp. gr. 1.42). The reaction is apt to be very rapid and the acid should be added in small portions to the covered beaker which is preferably kept cooled by immersion in ice water. Effect complete solution by gentle warming if necessary.

Add 30 cc. of HCl (sp. gr. 1.19) and evaporate to dryness on the steam-bath. Add 30 cc. more of HCl (sp. gr. 1.19), again evaporate to dryness, and bake for 30 minutes at 105° C. Moisten the residue with 30 cc. of HCl (sp. gr. 1.19) and evaporate on the steam-bath to a volume of 5 cc. Add 5 cc. of HCl (sp. gr. 1.19), dilute to 150 cc. and filter. Wash with warm water.

Adjust the volume of the solution to 250 cc. which should now contain about 3 to 4 per cent by volume of HCl. Warm to 75° C., add 30 cc. of $BaCl_2$ (10-per-cent), and allow to stand for 24 to 40 hours.

Filter, with as little transfer of the precipitate as possible, through a paper of close texture. Wash the paper two or three times with cold 1-per-cent HCl and seven times with warm water. Ignite the paper and contents in a platinum crucible. Treat the ignited residue with 5 cc. of HCl (sp. gr. 1.19) and transfer the resulting solution to the original beaker containing most of the precipitate of $BaSO_4$. Digest the combined portions of $BaSO_4$ so as to dissolve any barium vanadate. Adjust to 150-cc. volume and 2-per-cent acidity, add 8 cc. of 10-per-cent $BaCl_2$ solution and allow to stand for 24 hours. Filter on a paper of close texture, wash three times with cold 1-per-cent HCl and 10 times with warm water. Ignite the paper in platinum, cool in a desiccator and weigh. A blank should be carried through all steps of the determination. The corrected weight of the ignited barium sulfate multiplied by three is equal to the percentage of sulfur.

NOTES

If desired, the washings can be kept separate and examined for $BaSO_4$ as follows: Evaporate the washings to dryness. Dissolve the slight residue in 50 cc. of hot dilute HCl (2:98), add 2 cc. of $BaCl_2$ solution (10-per-cent), digest at 70 to 80° C. for two hours, avoiding any undue evaporation. Filter on a small paper of close texture and wash as above. The recovery of $BaSO_4$, which is ordinarily obtained, represents approximately 0.001 to 0.002 per cent of sulfur.

In high-silicon alloys the residue from the initial acid digestion should be evaporated with hydrofluoric and nitric acids, fused with sodium carbonate and the water extract of the melt added to the main solution.

DETERMINATION OF SILICON
BY THE
NITRO-SULFURIC METHOD

SOLUTIONS REQUIRED

Nitro-Sulfuric Acid.—Pour 160 cc. of sulfuric acid, (sp. gr. 1.84) slowly and with stirring into 660 cc. of water, and add 180 cc. of nitric acid (sp. gr. 1.42).

METHOD

Dissolve 0.9344 g. of the sample passing a No. 100 sieve of low or medium-silicon ferro-vanadium in 40 cc. of the nitro-sulfuric acid in a 250-cc. porcelain or platinum dish and evaporate the solution until "copious" fumes of sulfuric acid are evolved.

Ferro-vanadium containing 4 per cent or more of silicon is not completely soluble in nitric acid and sulfuric acid. In this case, fuse 0.9344 g. of the alloy, thoroughly mixed with 8 g. of sodium peroxide, in a 30-cc. crucible of pure iron. Five minutes heating at a dull red heat after the contents of the crucible have melted will insure complete decomposition of the sample. Allow the fusion to solidify, but before it has entirely cooled tap the covered crucible on an iron plate which will loosen the contents in a solid cake. Transfer the melt to a 250-cc. porcelain casserole, rinse the crucible with warm water and cautiously add the solution to the covered casserole. Next add 150 cc. of sulfuric acid (20 per cent by volume) and evaporate to copious fumes of sulfuric acid. Cool the sulfuric acid solution obtained in either procedure, add 125 cc. of water and heat for a few minutes with frequent stirring until all salts are dissolved. Immediately filter on a 9-cm. paper. Wash the precipitate with cold dilute HCl (5:95) and cold water alternately to complete the removal of iron salts, and finally with hot water until free from acid. Transfer the filter to a platinum crucible, char the paper without inflaming and finally ignite the covered crucible over a blast lamp or in a muffle furnace at 1050 to 1100° C. for at least 15 minutes. Cool in a desiccator and weigh. Add sufficient dilute H_2SO_4 (1:1) to moisten the silica and then a small amount of hydrofluoric acid. Evaporate to dryness, ignite and weigh. The difference in weights divided by 2 and multiplied by 100 represents the percentage of silicon in the alloy.

DETERMINATION OF ALUMINUM BY THE ETHER-HYDROCHLORIC ACID METHOD[1]

Dissolve 2 g. of the sample in 25 to 30 cc. of dilute nitric acid (sp. gr. 1.135) (Note 1). When action ceases, add 5 cc. of hydrochloric acid and evaporate to dryness. Add 10 cc. of hydrochloric acid, evaporate to dryness and bake the residue at a moderate temperature. Dissolve the residue in 10 cc. of hydrochloric acid, add 30 cc. of water, boil and filter. Wash the residue with hot dilute hydrochloric acid (1:9) followed by water (Note 2).

Evaporate the solution to dryness on a steam-bath, add 20 cc. of hydrochloric acid and warm gently until complete solution is obtained. Cool the solution in ice water and pass hydrochloric acid gas into it to saturation. Add 20 cc. of ether and continue passing the gas into the cold solution until it is again saturated. Allow the mixture to stand for several hours, keeping it cold by immersing the container in ice water. If the solution becomes warm or if it is not allowed to stand, the separation of hydrated aluminum chloride is not quite complete.

Filter through asbestos (small Bückner-asbestos pad washed once with cold ether - hydrochloric acid mixture immediately before use), wash, by decantation, with a cold mixture of equal parts of strong hydrochloric acid and ether which has been saturated with hydrochloric acid gas. The aluminum chloride retains a little vanadium.

Dissolve the precipitated aluminum chloride in the least possible amount of dilute hydrochloric acid (1:9) back into the original beaker, from which all of the precipitate need not have been removed. Evaporate the solution to near dryness on a steam-bath, add 10 cc. of strong hydrochloric acid and pass hydrochloric acid gas into the solution to saturation, add 15 cc. of ether and continue passing hydrochloric acid gas to saturation, keeping the solution cold by means of ice water. Let stand several hours, filter on asbestos and wash with the ether - hydrochloric acid mixture as before.

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[1] Gooch-Havens, *Am. Journal of Science*, Vol. 4, pp. 11–416.

Dissolve the aluminum chloride in dilute hydrochloric acid (1:9) into a 250-cc. beaker, add a few grams of ammonium chloride, make faintly alkaline with ammonia, boil, filter (using paper pulp), wash with hot water, dry, ignite and weigh as Al_2O_3.

According to published data, this method gives a complete separation of aluminum from iron, beryllum, zinc, copper, mercury and bismuth. A little vanadium is carried down in the first separation, but two chloride precipitations seem to give a practically complete separation. The proportion of vanadium retained in the final alumina is hardly appreciable when working on a 35-per-cent vanadium alloy.

The effect of chromium is not known; presumably some of it would be included in the Al_2O_3. This could be corrected for by fusing the Al_2O_3 with sodium peroxide and estimating the Cr_2O_3 by color.

NOTES

1. Dilute nitric acid (sp. gr. 1.135) is recommended for solution. While this requires rather long digestion at steam-bath temperature, solution seems to be quite complete even when the alloy is high in silicon. In some cases where silicon is very high it is necessary to use a little hydrofluoric acid with the nitric acid. In this case solution should be effected in a platinum dish, the solution evaporated to dryness and the dry residue transferred to a beaker before treatment with hydrochloric acid. A little dilute hydrochloric acid may be used to loosen any oxides that adhere to the dish.

2. The siliceous residue may retain alumina; it should be ignited in platinum. Silica should be expelled by $HF + H_2SO_4$ and the residue fused with $KHSO_4$. After dissolving the fusion in water plus a little HCl, the liquid is made faintly alkaline with ammonia and boiled. Any iron and alumina precipitate is removed by filtration, washed with water, dissolved in a little hot dilute HCl and added to the solution of the alumina obtained after the first ether - hydrochloric acid separation.

FERRO-TUNGSTEN AND TUNGSTEN METAL

DETERMINATION OF TUNGSTEN

SOLUTIONS REQUIRED

Cinchonine Solution.—Dissolve 125 g. of cinchonine in a mixture of 500 cc. of HCl (sp. gr. 1.19) and 500 cc. of distilled water.

Cinchonine Wash Solution.—Dilute 30 cc. of the above solution to one liter.

Treat 1 g. of the material passing a No. 100 sieve in a large (60-cc.) platinum crucible with 5 cc. of HF; add HNO_3 drop by drop until the metal dissolves. Add 3 to 4 cc. of H_2SO_4 and evaporate on a steam-bath until $HNO_3 + HF$ is expelled. Shake gently over a small Bunsen flame, or heat slowly in a sheet iron "radiator" (described by Hillebrand)[1] until H_2SO_4 fumes strongly. Cool, transfer to a 250-cc. beaker with water, finally wiping the crucible with a little filter paper. A little WO_3 sticks to the crucible; it can not be removed by wiping. Reverse the crucible. Dilute the contents of the beaker to about 150 cc. with water, add 10 cc. of hydrochloric acid (sp. gr. 1.19) and boil for a few minutes. Remove from the source of heat, add 10 cc. of cinchonine solution and digest the solution at 80 to 90° C. with occasional stirring, for 30 to 45 minutes.

Add some paper pulp to the solution, stir the solution and when the tungsten precipitate has settled, filter on a 9-cm. paper containaing a little "ashless" filter pulp. Wash thoroughly with hot cinchonine solution and finally several times with warm 1-per-cent hydrochloric acid. Gently ignite the filter and residue of crude WO_3 in the crucible in which it was originally treated until the carbon is consumed. Add a few drops of nitric acid and dry in the radiator. Ignite the covered crucible for 5 minutes in the full heat of a Bunsen burner, cool, and weigh. Ignitions may be made at about 750° C. in an electric muffle. As WO_3 is slowly but steadily volatilized at temperatures above 750° C., serious error may result from continued ignition of the tungsten trioxide at the temperature that is specified. Add about 5 g. of Na_2CO_3 and fuse, running the fusion around the side of the crucible to remove all WO_3. Dissolve the fusion in hot water, add alchohol, heat, filter and wash with hot water. Place the filter in the crucible and ignite; add a little Na_2CO_3 and fuse again. Dissolve the fusion in water, filter and wash *very* thoroughly with hot water to remove the last traces of Na_2CO_3; ignite in the same crucible as at first, cool, and weigh. The difference between the weight of crucible plus residue and the weight of crucible plus impure tungstic oxide is $WO_3 \times 0.7931 = W$.

NOTES

At the best, direct determination of tungsten in high-grade metal is subject to inherent errors. Accuracy within 0.2 per cent is all that can be expected by any method.

DETERMINATION OF CARBON

See the Determination of Carbon in Ferro-Chromium by the Direct Combustion Method.[2] The alloy ground to pass a No. 100 sieve and mixed with 2 g. of pure iron is burned in an electric furnace heated at 1050 to 1100° C. A "blank" on the ingot iron must be determined and deducted.

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[1] U. S. Geological Survey *Bulletin No. 700*, p. 33.

[2] See p. 553.

DETERMINATION OF PHOSPHORUS

Treat 1 g. of the finely powdered sample in a platinum dish, fitted with a gold cover, with 15 cc. of HNO_3 (sp. gr. 1.42), add 3 cc. of HF and warm gently. When action subsides add 3 cc. more of HF. After action subsides, boil, remove cover and if decomposition is not complete add more HF and boil again. When solution is complete, wash off the cover and evaporate at a low heat to a volume of about 10 cc., then add 3 or 4 drops of concentrated permanganate solution and continue evaporation until crusts of WO_3 begin to form at the edges, that is to a volume of about 6 cc. Add 5 cc. of H_2SO_4 and evaporate on the stove at a low heat until HF and HNO_3 are expelled and H_2SO_4 fumes are given off. (Strong heat causes spattering and also causes hard over-baked crusts to form on the bottom of the dish which resist subsequent treatment.) Cool, add 25 cc. of water and boil (by agitating over a Bunsen flame) until all soluble salts are dissolved. Discharge pink color, due to excess permanganate, by adding sulfurous acid drop by drop. The pink color may not be very evident but the SO_2 is added even so to reduce higher oxides of manganese. Boil for a minute or two after adding the SO_2. Add 1.20 g. of pure tartaric acid and, when this is dissolved and the solution is cooled to a temperature of about 50° C., add 20 cc. of NH_4OH (sp. gr. 0.90) diluted with an equal volume of water. The precipitated tungstic acid should dissolve completely, giving a clear solution. The solution becomes hot from the reaction between H_2SO_4 and NH_4OH. While it is still hot, add 10 cc. of magnesia mixture and transfer it from the platinum dish to a 6-oz. glass-stoppered bottle. Set the bottle in ice water and when it is thoroughly cooled add four or five glass beads about 6 mm. in diameter. Stopper it tightly and shake in an efficient shaking machine for at least ten minutes. The agitation should be violent. The beads aid in starting the formation of the magnesium precipitate; after agitation add 15 cc. of strong NH_4OH and return the bottle to the ice-water tank and put into a refrigerator to stand overnight. Phosphorus separates as magnesium ammonium phosphate free from tungsten but containing possibly basic magnesium compounds. After standing overnight filter the solution through a 9-cm. paper containing a little paper pulp and wash the bottle and paper thoroughly by small additions of ammonia wash water (1 part NH_4OH to 3 parts H_2O). Do not attempt to remove all the precipitate from the bottle but remove the beads to the filter. The precipitate consists of magnesium ammonium phosphate and arsenate together with silica and other impurities. Tin, tungsten, molybdenum, vanadium and titanium are eliminated by precipitation with magnesia mixture in the presence of tartaric acid. Dissolve the magnesia precipitate in hot dilute HCl and evaporate to a small volume, say 7 or 8 cc. Add 10 cc. of strong HCl and 0.5 g. of KBr and evaporate to dryness. Take up with 5 cc. of HNO_3 and evaporate to dryness again. The KBr serves to reduce As^{v} to As'''. The addition of strong HCl and boiling followed by evaporation to dryness eliminates As. Dissolve residue in 60 cc. of HNO_3 (sp. gr. 1.135), filter into a 250-cc. shaking bottle; add 40 cc. of NH_4OH (sp. gr. 0.96), cool to 33° C., add 30 cc. of molybdate solution (Blair) and finish the determination by the alkalimetric method (Handy) described in Blair's Chemical Analysis of Iron.

NOTES

The usual proportions of HNO_3 and HF are reversed in order to provide a constant excess of HNO_3 to oxidize P. The procedure given takes a little longer than when the sample is treated with HF first and HNO_3 is added a little at a time, but the solution is finally complete. It is necessary to keep the platinum dish covered after action begins, as the reaction is somewhat violent.

The platinum dishes recommended are:

8 cm. in diameter at top;
7.8 cm. in diameter at bottom;
4 cm. high.

They have flat bottoms and are wire-rimmed at the top to give additional stiffness. They weigh 58 to 60 g. each and hold about 175 cc. The covers are made of pure gold (for economy), "dished" like a crucible cover to fit the top of the dishes closely. The dishes have small lips to aid pouring. The "tongue" of the cover overlaps the lip. Ordinary round-bottomed dishes may be used, but the manipulation is much more difficult; there is a greater tendency to spattering and danger of local baking or overheating in evaporating to fumes of H_2SO_4. If the separated WO_3 is overheated locally it does not dissolve readily in NH_4OH. In flat-bottomed dishes, the WO_3 is spread in a thin layer and heat is applied evenly all over the bottom.

A good shaking machine should be used. The magnesia precipitate may be started by shaking the bottles by hand, but it is a tiresome job. The solution must be cold, say 8 or 10° C., when shaking begins.

Permanganate solution is added to insure complete oxidation of phosphorus, as in steel analysis. The color of the permanganate gradually fades in the hydrofluoric nitric solution, but after evaporating to fumes and adding water the solution is usually slightly pink.

The amount of tartaric acid is limited to 1.20 g. since ammonium tartrate retards the formation of the magnesia precipitate. Complete precipitation can only be obtained by brisk agitation and by keeping the solution very cold, followed by long standing in a cold place. By this method determinations started at 3 p. m. may be completed by noon the next day.

DETERMINATION OF SULFUR AND SILICON

SOLUTIONS REQUIRED

Cinchonine Solution.—Thoroughly wash cinchonine reagent with hot water on a Büchner funnel, dry, and dissolve 125 g. of the washed cinchonine in a mixture of 500 cc. of HCl (sp. gr. 1.19) and 500 cc. of distilled water.

Cinchonine Wash Solution.—Dilute 30 cc. of the above solution to one liter.

Barium Chloride Solution.—One hundred grams of $BaCl_2 \cdot 2H_2O$ to 1000 cc.

SULFUR

Fuse 2 g. of the finely powdered metal, intimately mixed with 8 g. of dry Na_2CO_3 and 2 g. of powdered KNO_3, in a large platinum crucible (40 cc.). The fusion is best done over an alcohol blast flame or in an electric muffle to avoid contamination with sulfur contained in illuminating gas. Leach the fusion with 100 cc. of distilled water in a 250-cc. beaker. Filter into a 12-cm. porcelain casserole and wash the residue several times with hot distilled water. The solution contains practically all of the sulfur and most of the tungsten and silica. The residue contains some of each, but not more than traces of sulfur.

Render the liquid acid with 20 cc. of HCl and evaporate to dryness on a steam-bath, thus precipitating tungstic acid. After the residue is dry, heat it to 110 to 120° C. in an oven to dehydrate silica, take up with 5 cc. of HCl and 50 cc. of water, add 3 cc. of a strong cinchonine solution and boil, or heat just short of the boiling point, for 30 minutes. Let the solution stand for at least three hours and preferably overnight. By means of the cinchonine, all WO_3 is rendered insoluble. Filter through an 11-cm. filter paper (free from ash) which has been liberally treated with an emulsion of filter paper pulp and wash the residue with dilute cinchonine solution. Reserve the residue. The filtrate contains all the sulfur; heat it to boiling and add 10 cc. of barium chloride solution, boil for ten minutes, allow it to stand overnight, filter on a 9-cm. filter paper (free from ash), wash repeatedly but not excessively with hot water, ignite in platinum and weigh as $BaSO_4$. If the work is properly carried out every trace of WO_3 is removed from the solutions and there is no danger of including $BaWO_4$ with the $BaSO_4$. If one suspects that the $BaSO_4$ is not quite pure, it should be fused with a little Na_2CO_3, leached with water and filtered; the filtrate should be acidulated with HCl, a few drops of cinchonine solution added and the solution evaporated to dryness on a steam-bath. The dry residue should be dissolved in a little water, a few drops of HCl added, and any residue filtered off. In the clear filtrate $BaSO_4$ should be again precipitated as before. The weight of $BaSO_4 \times 0.1374 = S$.

"Blanks" on all reagents must be made parallel with the determination; this is important since all sodium carbonate obtainable contains sulfur and there is sometimes more or less of it in cinchonine. All evaporations should be made over steam and the operations conducted in a place free from sulfur gases.

NOTE

Evolution of sulfur in a stream of hydrogen saturated with hydrochloric acid as in Johnson's Method is optional.

SILICON

The residue of tungstic oxide from which the solution for the determination of sulfur was filtered contains most of the silica. The residue from leaching the original fusion contains the rest.

Wash the residue from Na_2CO_3 fusion from the filter paper with a fine jet of water into a small casserole, add an excess of HCl and evaporate to dryness on a steam-bath, heat the dry residue at 110 to 120° C. to dehydrate the silica, drench with hydrochloric acid, dilute to 100 cc. with hot water, heat until salts are in solution, and filter on a small filter paper (free from ash). Wash thoroughly with hot dilute HCl (1:100) and finally with hot water. Reserve the filter.

Wash the residue of tungstic oxide, silica and filter paper pulp from the filter into a small casserole, add 5 cc. of HCl and heat for a few minutes, then filter again through the same paper and wash it *thoroughly* with hot water. This second washing is necessary to remove sodium salts completely. (The original washing sufficed to remove all of the small amount of sulfur present.) Transfer the well-washed filter and residue to a platinum crucible, add the small filter containing the little silica recovered from the Na_2CO_3 fusion residue and ignite both to constant weight in a platinum crucible. On account of the presence of filter paper pulp, the ignited residue is porous and friable; if paper pulp had not been used the WO_3 would be dense and not easily susceptible to the subsequent treatment. Cool the ignited residue of WO_3 and SiO_2 and weigh it. Add 2 or 3 drops of H_2SO_4 and 5 or 6 cc. of pure HF. Digest at a gentle heat for some time, and then slowly evaporate off the HF. When H_2SO_4 fumes are evolved, cool and again add HF, digest as before, evaporate off the HF, ignite gently to expel H_2SO_4 and heat strongly for ten or fifteen minutes. Cool and weigh. The loss in weight after expulsion is SiO_2 which multiplied by 0.4672 equals silicon.

TENTATIVE RECOMMENDED PRACTICE
FOR
CARBURIZING AND HEAT TREATMENT OF CARBURIZED OBJECTS[1]

Serial Designation: A 37 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope.

1. This recommended practice is intended solely for guidance, and is not to be construed as entering in any manner into specifications of the American Society for Testing Materials.

Purpose and General Procedure.

2. Carburizing is intended to increase the carbon content in the surface layers of low-carbon steel objects so they may be made hard and wear-resistant on the outside with a strong, tough, inner portion or core. This type of structure is suitable for parts which are required to stand considerable shock and friction. The process consists in heating the steel in contact with a carbonaceous material for a long time at temperatures above the critical temperature of the steel.

Factors Governing the Process.

3. The primary factors governing the process are:

(*a*) Chemical composition of the steel;
(*b*) The carburizing operation;
(*c*) The hardening operation.

Chemical Composition of Steel.

4. The steel should be selected according to the service requirements. A well-made carbon steel will be satisfactory for most ordinary purposes. The carbon content of simple and alloy steels should preferably be below 0.20 per cent. High manganese above 0.60 per cent is sometimes specified to obtain increased hardness and increased core strength. High phosphorus and sulfur contents improve the machineability but generally indicate a steel of poor quality. These elements are, however, often added intentionally to steels of good quality to improve their machining qualities.

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[1] Criticisms of this Recommended Practice are solicited and should be directed to Mr. J. H. Hall, Secretary of Committee A-4 on Heat Treatment of Iron and Steel, Taylor-Wharton Iron and Steel Co., High Bridge, N. J.

This Recommended Practice, when adopted as standard, will supersede the present Recommended Practice for Heat Treatment of Case-Hardened Carbon-Steel Objects (Serial Designation: A 37 – 14), 1924 Book of A.S.T.M. Standards.

In alloy steels, nickel tends to retard the grain growth at carburizing temperatures, depresses the critical temperature, and allows the steel to be hardened at lower quenching temperatures. Chromium is used alone in percentages up to 1.25 per cent and gives a greater abrasive hardness to the case. S. A. E. Steel No. 3115, containing about 1.25 per cent nickel and 0.60 per cent chromium is recommended for general high grade use. A molybdenum content of about 0.25 per cent or combined with about 1.75 per cent of nickel is used with good results. The use of molybdenum steel containing chromium is not recommended as this steel has an inherent tendency to distort in the carburizing operation.

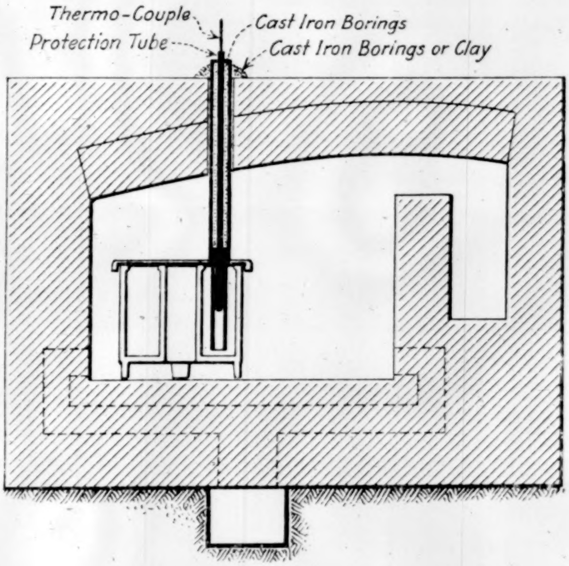

FIG. 1

5. Carburizing materials are divided into three general classes: solids, liquids, and gases, the solid type being generally used. The recommendations given apply to this type only. Commercial compounds are usually better than those made by the user. One part of new solid compound to three parts of used compound is recommended as a shop mixture. The used compounds should be screened to remove the dust. **Compounds.**

6. Packing pots are made from cast iron, cast and pressed steel and special heat-resisting alloys. The latter are recommended. Comparisons are usually made on a heat-hour-cost basis, the life of **Containers.**

the containers being determined by actual service and this figure divided into the cost of the pot which gives the cost per service-heat-hour. The proportions of the pot in relation to the parts to be carburized is of primary importance as well as the proper distribution of metal in the walls, reinforcement ribs, legs, etc. A compromise in size is found between the very small containers which are expensive in handling practice and the large containers which have a tendency to heat unevenly. The size of the pot should be governed by the size of the parts.

Temperature. 7. The effective temperature is the temperature inside the pot. The carburizing time should be designated as the number of hours at the carburizing temperature. The heating through of the pots is judged in two ways: by estimating with the eye, and by placing thermocouples inside the pot as shown in Fig. 1. The latter method is recommended except on pots which are so small that it would be impracticable to place a thermocouple inside them.

Pyrometers. 8. The use of recording pyrometers is recommended. Where the method shown in Fig. 1 is used, an extra couple should be placed in the furnace so that the furnace temperature may be more readily controlled, as otherwise excessive furnace temperatures or extremely variable heats may be obtained.

Heating. 9. Quick heating to the carburizing temperature will cause more distortion of the parts than slow heating. The temperature in any pot must be comparable with the temperature inside any other. To this end, the furnace must be designed to heat evenly and the pots must be spaced on the furnace hearth so that they will be heated evenly and uniformly.

Carburized Zone. 10. The carbon content at the surface of the work increases with the carburizing temperature. The formation of free carbide at the grain boundaries is undesirable, as it leaves a very brittle surface exposed to the service stresses. The surface carbon should not exceed 1.15 per cent. This will be reduced by the finish grinding operation. The carbon content is governed by the carburizing compound and the temperature while gradation depends upon the analysis of the steel and the rate of cooling. The allowable temperature depends upon the nature of the work. Temperatures of 941 to 954° C. (1725 to 1750° F.) may be used where speed and low expense are primary considerations. For general practice, 899° C. (1650° F.) is recommended. The carburizing temperature should be lowered about 38° C. (100° F.) when the regenerative quench is not to be used. Thin cases, of 0.010 to 0.015 in., are better obtained with a carburizing temperature of 843° C. (1550° F.).

TABLE I.—RECOMMENDED TEMPERATURES AND COOLING MEDIUMS TO BE USED FOR CARBURIZING AND HEAT TREATMENT OF CARBURIZED OBJECTS.

The temperatures and cooling mediums given in this table are dependent upon and must be varied according to the instructions given in the main section of the recommended practice. Particular attention should be given to varying the regenerative quench with the size of the part and the degree of distortion which must be avoided.

S. A. E. Steel No.	Composition						Preliminary Heat Treatments (use either A or B)									Carburizing			Refining and Hardening Treatments							
							Treatment A			Treatment B																
	Carbon, per cent	Manganese, per cent	Phosphorus, maximum per cent	Sulfur, maximum per cent	Nickel, per cent	Chromium, per cent	Normalizing Temperature		Cool in	Quenching Temperature		Cool in	Reheat to		Cool in	Temperature		Cool in	Regenerative Temperature		Cool in	Hardening Temperature		Cool in	Tempering Temperature	
							deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.
1020	0.15–0.25	0.30–0.60	0.045	0.05	....	....	900	1650	air	900	1650	oil	595	1100	air	900	1650	pots	871	1600	oil	775	1425	brine	205	400
1015	0.10–0.20	0.30–0.60	0.045	0.05	....	....	900	1650	air	900	1650	or	595	1100	air	900	1650	pots	871	1600	oil	775	1425	brine	205	400
1025	0.20–0.30	0.50–0.80	0.045	0.05	....	....	900	1650	....	.....	.....	water	595	1100	air	900	1650	pots	871	1600	oil	775	1425	brine	205	400
2320	0.15–0.25	0.50–0.80	0.04	0.015	3.25–3.75	....	925	1700	air	830	1525	oil	595	1000	air	900	1650	pots	829	1525	oil	745	1375	oil	165	325
2315	0.10–0.20	0.30–0.60	0.04	0.015	3.25–3.75	....	.....	.....	....	.....	.....	....	.....	.....	....	.....	.....	....	.....	.....	....	.....	.....	.....	.....	.....
2512	max. 0.17	0.30–0.60	0.04	0.015	4.50–5.25	....	900	1650	air	830	1525	oil	595	1100	air	885	1625	pots	830	1525	oil	745	1375	oil	165	325
3115	0.10–0.20	0.30–0.60	0.04	0.015	1.00–1.50	0.45–0.75	925	1700	air	870	1600	oil	595	1000	air	900	1650	pots	845	1550	oil	760	1400	oil	165	325
3120	0.15–0.25	0.30–0.60	0.04	0.015	1.00–1.50	0.45–0.75	925	1700	air	870	1600	water	595	1100	air	900	1650	pots	845	1550	oil	{760 {782	1400 1440	water oil	165 165	325 325
3215	0.10–0.20	0.30–0.60	0.04	0.040	1.50–2.00	0.90–1.25	925	1700	air	845	1550	oil	620	1150	air	900	1650	pots	845	1550	oil	774	1425	oil	165	325
3220	0.15–0.25	0.30–0.60	0.04	0.040	1.50–2.00	0.90–1.25	925	1700	air	845	1550	oil	620	1150	air	900	1650	pots	845	1550	oil	760	1400	oil	175	350
3312	max. 0.17	0.30–0.60	0.04	0.040	3.25–3.75	1.25–1.75	900	1650	air	830	1525	oil	650	1200	air	885	1625	pots	855	1575	oil	760	1400	oil	190	375
3325	0.20–0.30	0.30–0.60	0.04	0.040	3.25–3.75	1.25–1.75	....	.....	....	815	1500	oil	650	1200	air	885	1625	pots	855	1575	oil	760	1400	oil	190	375
3415	0.10–0.20	0.45–0.75	0.04	0.040	2.75–3.25	0.60–0.95	900	1650	air	845	1550	oil	620	1150	air	885	1625	pots	845	1550	oil	760	1400	oil	175	350
6120[a]	0.15–0.25	0.50–0.80	0.04	0.04	....	0.80–1.10	900	1650	air	900	1650	oil	595	1100	air	899	1650	pots	885	1625	oil	802	1475	water	190	375

[a] This steel is a chromium-vanadium steel, the vanadium requirement being 0.15 per cent minimum with a desired content of 0.18 per cent.

Measurement. 11. The "depth of case" shall be taken to be the depth of the refined case to be observed with the naked eye after carburizing, hardening at the temperature giving maximum case refinement, and breaking for fracture.

Regenerative Quench. 12. To obtain the best core condition, the part should be allowed to cool in the pots, outside the furnace, reheated and quenched from a temperature which will refine the core and break up any undesirable structures of cementite. This is called a regenerative quench.

TABLE II.—COMMON DEFECTS IN CARBURIZED OBJECTS.

Defect	Cause	Indication	Correction
Depth of case and carbon content outside specified limits.	A. Carburizing temperature not correct.		
	A1. Furnace temperature uneven. Temperature of containers not uniform.	Results vary in the same furnace and from the same containers.	Redesign furnaces. Modify heating practice. Allow longer time for bringing furnaces to temperature.
	A2. Faulty pyrometer readings.	Entire furnace charges unsatisfactory.	Check pyrometer system.
	A3. Carburizing compound too weak or too strong.	Results from entire department vary from usual results.	Check carburizing compound before accepting shipment. Change time and temperature until desired results are obtained.
Soft spots obtained in hardening.	B1. Improper hardening. Temperature low or uneven.	File test.	Raise hardening temperature
	B2. Improper quench.	File test.	Use faster quench, brine or spray.
	B3. Surface decarburized.	File test. Determine whether decarburization occurred in carburizing or hardening furnace.	Correct carburizing practice. Correct heating practice for hardening.
	B4. Low carbon content or uneven penetration.	As above for B3.	Correct carburizing practice. Try other compound. Check pyrometers.
Unsatisfactory fractures.	C1. Too high hardening temperatures.	Fracture test.	Decrease or adjust hardening temperature.
	C2. High surface carbon (freckled edges).	Fracture test.	Decrease hardening temperature. Use double quench with rather high temperature for regenerative quench. Use weaker compound.

Cooling of Pots. 13. The cooling of the carburized parts should take place in the containers, outside of the furnace. Where cost of production is vital, the parts may be quenched directly from the pot in either oil or water.

Grinding. 14. In determining the depth of case desired, sufficient allowance must be made for the metal which will later be removed by grinding. A tempering operation will tend to eliminate the tendency for grinding cracks to form.

Hardening. 15. The hardening temperature of the case is below the critical temperature of the core and the grain size produced in the core, by the carburizing temperature or regenerative quench, is not affected. The hardening temperature should be chosen as the temperature which gives the best fracture on several dummies or experimental pieces hardened from various temperatures. The figures given in Table I are the middle of the probable range of hardening temperatures.

16. The tendency to distort may be avoided by a preliminary heat treatment, either before or after the rough machining operation. The method of carburizing must be studied and the subsequent hardening operations carried on with extreme care as to the heating and cooling cycles. In many cases, the regenerative quench must be omitted as the high temperature of this operation causes too much warping. When omitted, the carburizing temperature should be lowered about 38° C. (100° F.). **Distortion.**

17. Tempering may be carried on at low temperatures, for the relief of strains, or at high temperatures, for the increase of toughness and uniformity without materially decreasing the hardness. An oil bath is preferable to an open furnace for temperatures up to 316° C. (600° F.). For simple carbon steels, a softening of the surface is found when the tempering temperature exceeds 190 to 204° C. (375 to 400° F.). For maximum hardness the temperature should be kept below this figure. **Tempering.**

18. Table II shows in condensed form the most common defects in carburized work together with the possible causes and methods for their identification and correction.

TENTATIVE SPECIFICATIONS
FOR
ALUMINUM BASE ALLOY SAND CASTINGS[1]

Serial Designation: B 26 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924; REVISED, 1925.

Material Covered.

1. These specifications cover commercial aluminum base alloy sand castings having a specific gravity of 3 and less.

I. MANUFACTURE

Process.

2. The alloy may be made by any approved method.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition.

3. The following alloys are described by these specifications; they shall conform to the following requirements as to chemical composition:

Alloy	Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Magnesium, per cent	Manganese, per cent	Zinc, per cent	Remarks
A.....	not under 96.5	1 to 1.5	not over 0.5	not over 0.5	trace	0.7 to 2.0	trace	
B.....	not under 92.5	not over 0.6	not over 1.0	4.5 to 6.0	trace	not over 0.2	not over 0.20	
C.....	not under 90.0	7 to 8.5	......	......	......	......	not over 0.20	Total of Fe, Si, Mn, Zn and Sn not to exceed 1.70 per cent.
D....	88 to 92	6 to 8.0	not over 1.50	......	......	......	not over 2.50	Total of Si, Mn, and Sn not to exceed 1.00 per cent.

Sampling.

4. (*a*) The sample for chemical analysis may be taken either by sawing, drilling or milling the casting or tension test specimen and shall represent the average cross-section of the piece.

(*b*) The saw, drill, cutter or other tool used shall be thoroughly cleaned. No lubricant shall be used in the operation, and the sawdust or metal chips shall be carefully treated with a magnet to remove any particles of iron introduced in taking the sample.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

These Tentative Specifications, when adopted as standard, will supersede the present Standard Specifications for Light Aluminum Casting Alloys (Serial Designation: B 26-21), 1924 Book of A.S.T.M. Standards.

III. PHYSICAL PROPERTIES AND TESTS

Tension Tests.

5. The alloys shall conform to the following minimum requirements as to tensile properties:

Alloy	Minimum Tensile Strength, lb. per sq. in.	Minimum Elongation in 2 in., per cent
A	18 000	8
B	16 000	3.5
C	18 000	..
D	19 000	..

Test Specimens.

6. (*a*) Two test bars shall be an integral part of large castings, or cast separately in the case of small castings to represent a lot or melt, and shall be molded in a manner similar to the castings which they represent. If the castings are heat treated, the test bars representing such castings shall be similarly heat treated.

(*b*) The manufacturer and purchaser shall agree whether the test bars can be attached to castings, on the locations of the bars, on the castings to which bars are to be attached, on the method of casting unattached bars and whether a "cast-to-size bar" or a "machined-to-size bar" shall be used.

(*c*) The "machined-to-size bar" shall be cast according to the dimensions shown in Fig. 1, and machined to conform to the dimensions shown in Fig. 2. The "cast to size" bar shall be cast according to the dimensions shown in Fig. 2 and shall be tested without machining. The ends shall be of a form to fit the holders of the testing machine in such a way that the load shall be axial.

Number of Tests.

7. (*a*) Tests shall as far as possible be made by heats or melts, but unless otherwise agreed, two tension tests shall be made upon each unit lot of 500 lb. or single delivery of less than 500 lb.

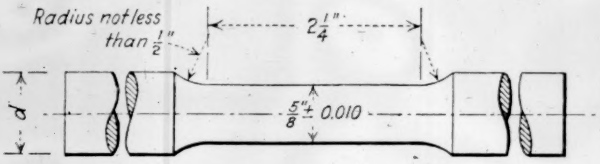
(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded; in which case the manufacturer and the purchaser or his representative shall agree upon the selection of another specimen in its stead.

IV. INSPECTION AND REJECTION

Inspection.

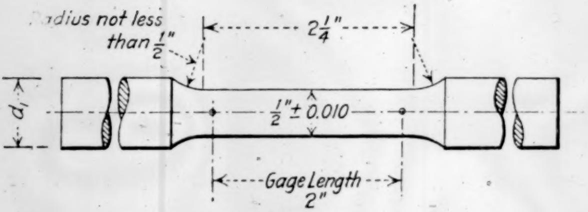
8. (*a*) Inspection may be made at the manufacturer's works where the castings are made, or at the point at which they are received, at the option of the purchaser.

(*b*) If the purchaser elects to have inspection made at the manufacturer's works, the inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.



NOTE:
The Gage Length, Parallel Section and Fillets shall be as shown, but the Ends may be of any Shape to permit of Machining according to the Requirements of Fig. 2.
It is recommended, that Diameter d be from $\frac{7}{8}$" to $1\frac{1}{8}$".

FIG. 1



NOTE:
The Gage Length, Parallel Section and Fillets shall be as shown, but the Ends may be of any Shape to fit the Holders of the Testing Machine in such a way, that the Load shall be Axial.
It is recommended, that Diameter d_1 be from $\frac{3}{4}$" to 1."

FIG. 2

Rejection. 9. Castings which show injurious defects revealed by machining operations subsequent to acceptance may be rejected, and if rejected, shall be replaced by the manufacturer free of charge to the purchaser. The full weight of the original material rejected shall be returned to the manufacturer.

EXPLANATORY NOTES

The Naval Gun Factory has been the largest user of Alloy A. Its resistance to salt-water corrosion makes it suitable for marine equipment. Although very large castings have been successfully poured from it, Alloy A is generally regarded as difficult to handle in the foundry. Its proportional limit is rather low.

Alloy B possesses very good foundry characteristics; the melt is quite fluid and the solidification shrinkage is low. It is especially suitable for the production of dense non-porous castings and of intricate shapes containing both heavy and thin sections. This alloy is the most resistant to corrosion of all the commonly used aluminum casting alloys. It is somewhat more difficult to machine than the other alloys and, as in the case of Alloy A, possesses a rather low proportional limit.

Alloys C and D are similar, both being substantially aluminum-copper alloys. The composition limits are such that secondary metal can be used in the manufacture of Alloy D. While no lower limits are placed on the iron and zinc in Alloy D, it is assumed they will be present and they are stated to improve the machining qualities of the castings.

The pattern maker's shrinkage is usually taken as 0.156 in. per foot for all these alloys. The weight lies within the range of 0.096 lb. per cu. in. for Alloy B, and 0.103 lb. per cu. in. for Alloy C, corresponding to the specific gravities 2.66 and 2.85, respectively. These are approximate values, since the weights will vary with the actual compositions of the individual samples.

Section 5.—The elongations of Alloys C and D are usually so low as to present difficulties in their accurate measurement. The fact that a very large percentage of all aluminum castings produced in this country is made from these alloys, is evidence that the low elongation is not indicative of excessive brittleness.

Section 6.—Some latitude has been allowed in the matter of test specimens; test bars "cast to size" are stronger than those which are "machined to size" and the tensile properties specified are regularly obtained in commercial production only with the "cast to size" bar. When "machined to size" specimens are required to meet the values given in these specifications, rejections may be expected to run higher than those experienced in usual commercial production. Owing to insufficient information being available on the tensile properties of "machined to size" specimens it was not possible to include a separate table of values for this type of specimen.

It is strongly recommended that a self-centering form of tension test specimen holder be used. Most aluminum alloys are brittle and give lower and less uniform test results when tested in ordinary wedge grips without centering.

TENTATIVE SPECIFICATIONS
FOR
BRONZE TROLLEY WIRE[1]

Serial Designation: B 9 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925.

I. MANUFACTURE

Scope. 1. These specifications cover round and grooved bronze trolley wire.

Material. 2. The material used shall be bronze of such nature and composition as to secure by proper treatment the qualities hereinafter specified for the finished wire. The copper used shall be electrolytic or low-resistance lake copper conforming in quality and purity to the requirements of either the Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars (Serial Designation: B 5) or of the Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars (Serial Designation: B 4) of the American Society for Testing Materials.[2]

Bronze of two qualities may be required:

(*a*) High Strength.

(*b*) Medium Strength.

II. PHYSICAL PROPERTIES AND TESTS

(*A*) *Round Wire.*

Tensile Strength and Elongation. 3. (*a*) Round wire shall be so drawn that its tensile strength and elongation shall not be less than the values given in Table I.

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[1] These specifications were prepared by Committee B-1 on Copper Wire in cooperation with a committee of the American Electric Railway Engineering Association. The specifications of the two organizations are identical save with respect to the requirements for the twist test, Section 4. Further cooperative work with respect to these requirements is being undertaken.

Criticisms of these Tentative Specifications are solicited and should be directed to Mr. J. A. Capp, Chairman of Committee B-1 on Copper Wire, General Electric Co., Schenectady, N. Y.

These tentative specifications, when adopted as standard, will supersede the present Standard Specifications for High-Strength Bronze Trolley Wire, Round and Grooved (Serial Designation: B 9 – 16), 1924 Book of A.S.T.M. Standards.

[2] 1924 Book of A.S.T.M. Standards.

The elongation shall be determined as the permanent increase in length due to the breaking of the wire in tension measured between bench marks placed upon the wire originally 10 in. apart. The fracture shall be between the bench marks and not closer than 1 in. to either bench mark.

(*b*) Tests upon a section of wire containing a braze shall show at least 95 per cent of the tensile strength of the unbrazed wire.

Elongation tests shall not be made on test sections including brazes.

Twist Test.

4. For the purpose of determining and developing defects which may be prejudicial to the life of trolley wire, owing to its peculiar service, as compared to that of copper wire for other purposes, round wire shall be subjected to the following twisting test. Three twist tests shall be made upon samples 10 in. in length between the holders of the machine. The twisting machine shall be so constructed that there is a linear motion of the tail stock with respect to the head. The twist shall be applied not faster than 10 turns per minute. All three samples shall be twisted to destruction and shall not reveal under test any seams, pits, slivers, or surface imperfections of sufficient magnitude to indicate inherent defects or imperfections in the wire. At the time of fracture the wire shall be twisting with reasonable uniformity. Wire shall not be considered satisfactory which does not withstand at least twenty turns before breaking.

TABLE I.—VALUES FOR TENSILE STRENGTH AND ELONGATION OF ROUND WIRE.

		HIGH-STRENGTH BRONZE WIRE		MEDIUM-STRENGTH BRONZE WIRE	
DIAMETER, IN.	AREA, CIRCULAR MILS	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION IN 10 IN., PER CENT	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION IN 10 IN., PER CENT
0.548.....	300 000	70 000	4.50	60 000	4.50
0.460.....	211 600	73 000	3.75	62 000	3.75
0.410.....	168 100	76 000	3.25	64 000	3.25
0.365.....	133 225	79 000	2.80	66 000	2.80
0.325.....	105 625	81 000	2.40	69 000	2.40

Resistivity.

5. Electric resistivity shall be determined upon fair samples by resistance measurements at a temperature of 20° C. (68° F.).

High-strength wire shall not exceed in resistivity 0.38320 ohms per meter gram. Medium-strength wire shall not exceed in resistivity 0.23574 ohms per meter gram.

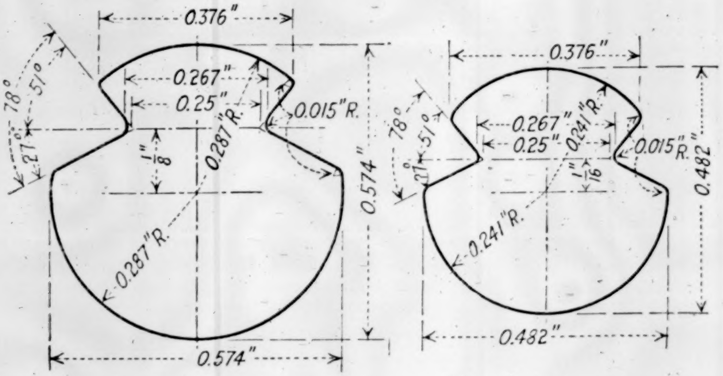
The values given above are based on bronzes of copper-tin compositions and represent classes of such wire formerly referred to as 40-per-cent and 65-per-cent conductivity wire.

Lower resistivities in wires meeting the physical qualifications of these specifications may be obtained by the use of other alloys.

(B) Grooved Wire.

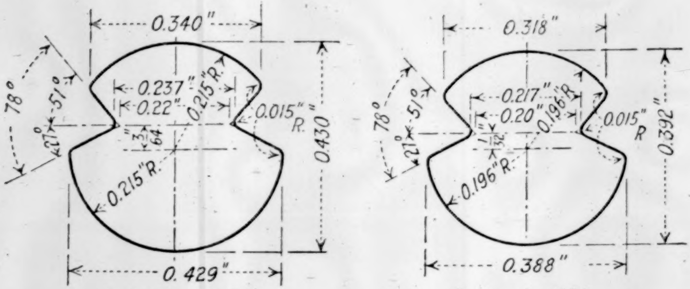
Physical Properties and Tests.

6. (*a*) The physical tests for grooved wire shall be made in the same manner as those upon the round wire. The tensile strength of grooved wire shall be at least 95 per cent of that required for round wire of the same nominal cross-sectional area; the elongation shall be the same as that required for round wire of the same nominal cross-sectional area. The twist test shall be omitted.


0.376"
78°
51°
27°
0.267"
0.25"
0.287" R.
0.015" R.
1/8"
0.574"
0.287" R.
0.574"
0.376"
78°
51°
27°
0.267"
0.25"
0.241" R.
0.015" R.
1/16"
0.482"
0.241" R.
0.482"

Grooved Wire
Nominal Cir. Mils, 300 000
Actual Area, 0.2356 sq. in.
Calculated Weight, 4786 lb. per mile

Grooved Wire
Nominal Cir. Mils, 211 600
Actual Area, 0.1665 sq. in.
Calculated Weight, 3382 lb. per mile


0.340"
78°
51°
27°
0.237"
0.22"
0.215" R.
0.015" R.
3/64"
0.430"
0.215" R.
0.429"
0.318"
78°
51°
27°
0.217"
0.20"
0.196" R.
0.015" R
1/32"
0.392"
0.196" R.
0.388"

Grooved Wire
Nominal Cir. Mils, 168 100
Actual Area, 0.1314 sq. in.
Calculated Weight, 2669 lb. per mile

Grooved Wire
Nominal Cir Mils, 133 225
Actual Area, 0.1083 sq. in.
Calculated Weight, 2201 lb per mile

FIG. 1

(*b*) Tests upon a section of wire containing a braze shall show at least 95 per cent of the tensile strength of the unbrazed wire.

Elongation tests shall not be made on test sections including brazes.

7. The requirements for resistivity of grooved wire shall be the same as those for round wire of the same nominal cross-sectional area. **Resistivity.**

III. DIMENSIONS AND PERMISSIBLE VARIATIONS

(*A*) ***Round Wire.***

8. (*a*) The size shall be expressed as the diameter of the wire in decimal fractions of an inch, using not more than three places of decimals, *i. e.*, in mils. **Dimensions and Permissible Variations.**

(*b*) Wire shall be truly cylindrical in form. The diameter shall not vary more than 1 per cent over or under the diameter specified.

(*B*) *Grooved Wire.*

9. Standard sections of grooved trolley wire shall be those known as the "American Standard Grooved Trolley Wire Sections" (the Standard Design of the American Electric Railway Engineering Association), shown in Fig. 1. **Sections.**

10. (*a*) Size shall be expressed as the nominal area of cross-section in circular mils, the standard sizes being as follows: **Dimensions and Permissible Variations.**

NOMINAL AREA, CIRCULAR MILS	ACTUAL AREA, SQ. IN.	CALCULATED WEIGHT BASED ON ACTUAL AREA, LB. PER MILE
300 000	0.2356	4786
211 600	0.1665	3382
168 100	0.1314	2669
133 225	0.1083	2201

(*b*) The weight of grooved trolley wire shall not vary more than 4 per cent over or under the weights specified in Paragraph (*a*).

IV. FINISH

11. (*a*) The wire shall be of uniform size, shape, and quality throughout, and shall be free from all scale, flaws, splits and scratches not consistent with the best commercial practice. **Finish.**

(*b*) Necessary brazes in trolley wire shall be made in accordance with the best commercial practice.

V. PACKING

12. All wire shall be shipped on substantial reels, suitable for the weight of the wire handled, and shall be well protected from injury. If reels are to be lagged, it shall be so specified by the purchaser. **Packing and Shipping.**

The ends shall be securely fastened to the sides of the reels with no less than six staples. The staples shall be at least 2 in. in length and made from not less than 0.145-in. diameter wire. Care shall be exercised in stapling not to damage the surface of the exposed layer of wire. All reels shall have the arbor holes reinforced with steel plates. Reels shall be marked legibly and indelibly with a serial number, size, kind, length and weight of wire, and such other information as is desired by the purchaser. The length or weight of wire to be wound upon reels shall be agreed upon in placing individual orders.

VI. INSPECTION AND REJECTION

Inspection. 13. All tests governing the acceptance or rejection of the wire, unless otherwise specified, shall be made at the place of manufacture with apparatus furnished by the manufacturer and in the presence of the purchaser, or his representative, who shall be furnished a copy of the tests. Every facility shall be given the purchaser, or his representative, to satisfy him as to the reliability of the results before the wire is delivered. If the purchaser waives inspection, and if he so elects at that time, he shall be furnished with a certified copy of tests made by the manufacturer.

Rejection. 14. Any reel of wire which fails to meet the requirements hereinbefore specified may be rejected. Failure of 30 per cent of the number of reels ready for inspection at one time shall be deemed sufficient cause for the rejection of the whole lot.

TENTATIVE SPECIFICATIONS
FOR
MUNTZ METAL CONDENSER TUBE PLATES[1]

Serial Designation: B 57 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Material Covered. 1. These specifications cover rolled Muntz metal plates for surface condenser tube sheets.

I. MANUFACTURE

Process. 2. The plates shall be made by hot rolling from castings of suitable dimensions.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition. 3. The plates shall conform to the following requirements as to chemical composition:

Copper	58.00 – 61.00 per cent
Lead	0.35 – 0.90 "
Iron	not over 0.15 "
Tin	not over 0.25 "
Other Impurities	not over 0.10 "
Zinc	Remainder

Chemical Analysis. 4. Analysis may be made on each lot of 5000 lb. or less.

Sampling. 5. The sample shall be taken by drilling or milling representative plates in such a way that a sample of the entire thickness shall be obtained. Equal quantities from each plate shall be thoroughly mixed. Samples so prepared shall be divided into three equal parts, one for the seller, one for the purchaser, and one for an umpire, if necessary.

III. PHYSICAL PROPERTIES AND TESTS

Tension Tests. 6. The material shall conform to the following minimum requirements:

TENSILE STRENGTH, LB. PER SQ. IN	YIELD POINT, LB. PER SQ. IN	ELONGATION IN 2 IN., PER CENT
50 000	20 000	35.0

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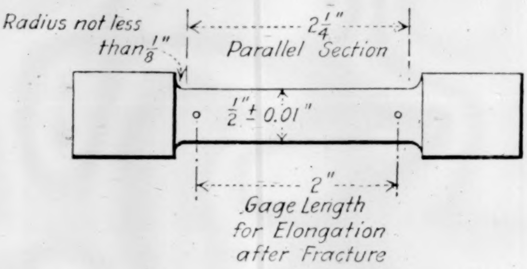
[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

Bend Test. 7. The bend test specimen shall stand being bent cold through 120 deg. around a pin, the diameter of which is equal to twice the thickness of the specimen, without cracking on the outside of the bent portion.

Tension Test Specimens. 8. Tension test specimens may be 1 in. in width and of the full thickness of the plate as rolled, or may be machined to conform to the dimensions shown in Fig. 1, in which case the ends shall be of a form to fit the holders of the testing machine in such a way that the load shall be axial.

Bend Test Specimens. 9. Bend test specimens shall be 0.5 by 1 in. in section with corners rounded to a radius not over $\frac{1}{16}$ in. In the case of thin plates, the specimen may have the same thickness as the plate, with corners rounded to a radius equal to one-fourth of the thickness of the plate.


Radius not less than $\frac{1}{8}$"
$2\frac{1}{4}$"
Parallel Section
$\frac{1}{2}$" ± 0.01"
2"
Gage Length for Elongation after Fracture

Note:-The Gage Length, Parallel Section, and Fillets shall be as Shown, but the Ends may be of any Shape to fit the Holders of the Testing Machine in such a Way that the Load shall be axial.

FIG. 1

IV. PERMISSIBLE VARIATIONS IN DIMENSIONS

Permissible Variations. 10. The thickness shall not vary from that specified by more than plus 3 per cent or minus 2 per cent.

V. WORKMANSHIP AND FINISH

Finish 11. The plates shall be free from injurious defects and shall have a smooth clean surface. The fracture shall be uniform in color and grain throughout.

VI. INSPECTION AND REJECTION

Inspection. 12. The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications.

Rejection 13. Material which fails to conform to these specifications will be rejected and the manufacturer shall be notified.

TENTATIVE SPECIFICATIONS
FOR
ALUMINUM INGOTS FOR REMELTING[1]

Serial Designation: B 24 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1918; REVISED, 1919, 1920, 1922, 1925.

Material Covered.

1. These specifications cover three grades of Virgin Aluminum, as follows:

GRADE.	ALUMINUM, PER CENT.
99.5	99.5 (Minimum)
99.0	99.0 (Minimum)
98.0	98.0 (Minimum)

Impurities.

2. The presence in any of the above grades of manganese, magnesium, zinc, calcium or similar metals commonly used in the light alloys, is not admissible and such ingot cannot be classed as Virgin Aluminum under these specifications. Copper is an allowable impurity up to a maximum of 0.1 per cent in the 99.5 grade; 0.25 per cent in the 99.0 grade; and 0.45 per cent in the 98.0 grade. Iron and silicon are also allowable impurities. The sum total of the impurities, however, shall not exceed 0.5 per cent in the 99.5 grade, 1.0 per cent in the 99.0 grade, nor 2.0 per cent in the 98.0 grade.

I. MANUFACTURE.

Manufacture.

3. No scrap shall be used other than that resulting in the manufacturer's own plant from the production of material of composition similar to that specified.

II. PHYSICAL DEFECTS.

Physical Defects.

4. Aluminum ingots shall have a clean surface and shall be free from dirt, slag and foreign material.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

III. SAMPLING.

Sampling.

5. If the aluminum is shipped in carload lots, then random samples shall be taken, but not less than five ingots for each car. If the shipment is in less than carload lots, one ingot shall be taken for each 10,000 lb. (4536 kg.) or fractional part thereof.

When it is deemed necessary, a sample may be taken from each heat of 500 lb. (226.8 kg.) or more of metal.

Drilling.

6. The ingots selected shall be drilled without lubricant completely through, or half way through from top and bottom. The drillings shall be fine, thin chips. They should be thoroughly mixed and the sample for analysis should not be less than 60 g.

IV. ANALYSES.

Analyses.

7. The chemical analysis shall be made in accordance with the Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (Serial Designation: B 40 – 25 T) of the American Society for Testing Materials.[1]

V. MARKING.

Marking.

8. The maker's brand and the grade of aluminum shall be cast on each ingot. When this is not practicable, the grade shall be indicated by spotting each large ingot, or one ingot in each bundle of small ingots, with a designating color of paint: white indicating the 99.5 grade, blue, the 99.0 grade, and red, the 98.0 grade.

VI. CLAIMS.

Claims.

9. Claims, to be considered, shall be made in writing within thirty days of receipt of material at the purchaser's plant, and the results of the purchaser's tests shall be given. The shipper shall within one week of receipt of such claim, either agree to satisfy the claim or send a representative to the purchaser's plant to resample the shipment, as specified in Section 3. Samples so taken shall be sealed and submitted to a mutually agreeable umpire, whose determination shall be final.

Settlement of Claims.

10. The expense of the umpire analysis shall be paid by the loser, or divided in proportion to the concession made in case of a compromise. In case of rejection being established, the damage shall be limited to the payment of freight both ways by the manufacturer for the substitution of an equivalent weight of metal meeting these specifications.

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[1] See p. 590.

TENTATIVE SPECIFICATIONS
FOR
NON-FERROUS INSECT SCREEN CLOTH[1]

Serial Designation: B 50 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923; REVISED, 1925.

Scope.

1. These specifications cover insect screen cloth woven from non-ferrous wire and intended to be used as a protection against insects or other small animals.

I. MANUFACTURE

Manufacture.

2. There shall be two classes of standard insect screen cloth depending upon the material used as follows:

(*a*) *Class A*, comprising cloth made from copper wire.
(*b*) *Class B*, comprising cloth made from a 90 copper-10 zinc alloy wire.

3. Insect screen cloth shall be manufactured in four weights or sizes of mesh as follows:

14-mesh
16-mesh
16-mesh heavy
18-mesh

4. The same quality and size of wire shall be used for both the warp and filling.

II. CHEMICAL PROPERTIES

Chemical Requirements.

5. The composition of the wire used for Class A insect screen cloth shall conform to the requirements either of the Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (Serial Designation: B 4), or of the Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (Serial Designation: B 5) of the American Society for Testing Materials.[2]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. W. H. Bassett, Secretary of Committee D-14 on Screen Wire Cloth, American Brass Co., Waterbury, Conn.

[2] 1924 Book of A.S.T.M. Standards.

6. The wire for Class B insect screen cloth shall conform to the following chemical requirements:

Copper	90.00 to 92.00 per cent
Lead, maximum	0.03 per cent
Iron, maximum	0.06 per cent
Zinc	Remainder

III. PHYSICAL PROPERTIES OF WIRE

7. The physical properties for all classes of screen wire shall be subject to agreement between the producer of the wire and the purchaser.

NOTE.—Wire for weaving insect screen cloth is usually furnished with a drawn temper. The needs of different weavers of wire cloth apparently call for difference in drawn temper and it is consequently at present impossible to give definite physical properties which would be satisfactory to the entire trade. It has, therefore, seemed better to leave this matter to specific agreement between the producers of wire and the manufacturers of wire cloth.

IV. STANDARD SIZES, WEIGHTS, DIMENSIONS, ETC.

Size of Wire.

8. (*a*) The nominal diameter of wires to be used for the various weights of standard insect screen cloth shall be in accordance with the following:

MESH	DIAMETER, IN.
14	0.0119
16	0.0113
16 (Heavy)	0 0150
18	0 0106

(*b*) The permissible variation from the nominal diameter of any of the wires specified above shall be ±0.00025 in.

9. The mesh as determined by counting the openings per linear inch for a length of one foot on any samples selected at random shall not vary from the nominal value in the warp, but an average variation of not more than $\frac{1}{2}$ mesh per linear inch shall be permissible in the filling.

10. Insect screen cloth shall be furnished in rolls of approximately 100 linear feet. At least 87.5 per cent of the rolls ordered shall be in one continuous length; the remaining rolls may be in two lengths.

11. The widths of standard insect screen cloth shall be from 18 to 48 in. in multiples of 2 in. The permissible variation in over all width as measured from outside wire to outside wire shall be $\pm\frac{1}{8}$ in.

V. WORKMANSHIP AND FINISH

12. (*a*) The insect screen cloth shall be free from all imperfections and blemishes not consistent with best commercial practice.

(*b*) Well-made splices in individual wires are permitted at any point in the screen cloth.

VI. INSPECTION

13. (*a*) All testing and inspection, both of individual wires and completed cloth, shall be made at the place of manufacture. Tests on individual wires shall be made on samples taken before weaving and not on wires removed from finished cloth.

(*b*) The manufacturer shall afford the inspector representing the purchaser all reasonable facilities to satisfy him that the material conforms to these specifications.

APPENDIX

The following table giving the weights of various mesh of insect screen cloth is not a part of these specifications, but is appended as a source of information for the purchaser:

MESH	APPROXIMATE WEIGHT PER 100 SQ. FT. OF INSECT SCREEN CLOTH WRAPPED AND LABELED READY FOR SHIPMENT
14	14½ lb.
16	15 "
16 (Heavy)	27¾ "
18	14 "

TENTATIVE METHODS OF CHEMICAL ANALYSIS OF ALUMINUM AND LIGHT ALUMINUM ALLOYS.[1]

Serial Designation: B 40 – 25 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921; Revised, 1923, 1925.

The methods of analysis listed under "Analysis of Aluminum" contemplate the analysis of aluminum containing more than 98 per cent aluminum and containing only silicon, titanium, iron, copper, carbon and traces of manganese as impurities.

Unless it is positively known that such elements as tin, antimony, zinc, lead, nickel, magnesium, sodium, nitrogen or oxygen (as aluminum oxide) are absent, the methods listed under the "Analysis of Light Aluminum Alloys" should be employed.

ANALYSIS OF ALUMINUM.

(Not less than 98 per cent Aluminum.)

DETERMINATION OF SILICON AND TITANIUM.

DETERMINATION OF TOTAL SILICON.

Solutions Required.

"*Acid Mixture.*"—1200 cc. of H_2SO_4 (25-per-cent by volume), 600 cc. HCl (sp. gr. 1.20) and 200 cc. HNO_3 (sp. gr. 1.42).

25-per-cent Sulfuric Acid.—Pour 250 cc. of H_2SO_4 (sp. gr. 1.84) into distilled water, and dilute to 1000 cc.

Method.

Dissolve 1 g. of the well-mixed sample in 35 cc. of the acid mixture in a covered flat-bottomed porcelain dish or casserole. When the drillings are completely dissolved, boil the solution to complete expulsion of HNO_3 and HCl and heat until copious fumes of H_2SO_4 have been given off for five minutes. Cool and take up the residue with 10 cc. of 25-per-cent H_2SO_4 and 100 cc. of water. Warm to complete solution of the sulfates and filter on an ashless filter paper. Wash the residue with hot water until free from sulfates and ignite in a platinum crucible. Fuse the residue with 8 to 10 times its weight of Na_2CO_3 and treat the fused mass in a casserole or beaker with 20 cc. of H_2SO_4 (1 : 3), washing out the crucible. Evaporate the solution until it fumes strongly to separate the SiO_2. Cool, dilute to about 100 cc., boil till the salts are dissolved, filter, wash well, ignite in a platinum crucible and weigh. Treat the ash with HF and a few drops of H_2SO_4 (1 : 3), carefully evaporate till dry, ignite and again weigh.

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[1] Criticisms of these Tentative Methods are solicited and should be directed, preferably before January 1, 1926, to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

From the difference between the two weights deduct a blank obtained from the reagents and filter. The rest of the loss in weight represents SiO_2 corresponding to the Si in the metal.

$$SiO_2 \times 0.4672 = Si.$$

NOTES.

Blanks should be carried along with all reagents and the proper corrections made.

In routine analysis, fusion of the first H_2SO_4 residue with sodium carbonate and subsequent treatment to the point of treating the ash with HF may be omitted. The first residue may be weighed and treated with HF, etc., as directed for the second H_2SO_4 residue. This, however, should not be done if the material contains much graphitic silicon or if the residue after ignition is dark colored.

DETERMINATION OF TITANIUM.

APPARATUS AND SOLUTIONS REQUIRED.

Nessler tubes or some form of colorimeter.

Hydrogen Peroxide U. S. P.—This reagent must be free from HF.

Standard Titanium Solution.—This solution is best prepared and standardized as follows:[1]

"Potassium titanium fluoride, K_2TiF_6, best serves as the starting point for the preparation of the standard solution. A quantity of this is recrystallized from boiling water one or more times, dried, and preserved in a glass-stoppered bottle. Enough of it to make 0.5 to 1 liter of the standard sulfate solution is put into a platinum dish and evaporated several times with strong sulfuric acid without bringing to dryness, till the fluorine is completely expelled. The residue is then taken up with water containing enough sulfuric acid to make at least

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[1] W. F. Hillebrand, "The Analysis of Silicate and Carbonate Rocks," *Bulletin No. 700*, U. S. Geological Survey, p. 157.

5 per cent of the latter when fully diluted. Two 50 to 100-cc. portions of the prepared solution are then further diluted, boiled, and precipitated with ammonia. The precipitates are collected on paper, washed with hot water till free from alkali, ignited moist in the filter, blasted, and weighed. Duplicates should agree almost exactly. From the weights found, the strength of the standard is calculated and the result affixed to the bottle containing it. The stopper of the bottle should be coated with vaseline and the needed quantities of solution should be withdrawn by a pipette, never poured. In a solution so prepared there is not enough alkali sulfate to weaken the color when peroxidized."

METHOD.

See "Determination of Total Silicon."

Fuse the non-volatile residue remaining after the silicon determination in a small amount of $K_2S_2O_7$, take up in a little 5-per-cent H_2SO_4 and add to the filtrates and washings from the silicon determination. Evaporate the solution to approximately 100 cc., and treat with 5 cc. H_2SO_4 (sp. gr. 1.84) and 3 g. of iron-free zinc. Heat until the zinc is nearly dissolved and the reduction of copper is complete. Decant the solution into another beaker, wash the zinc and copper with hot water and continue the evaporation of the solution to approximately 75 cc. Cool and transfer to a 100-cc. Nessler's comparison tube, add 5 cc. of 3-per-cent H_2O_2 and dilute to the 100-cc. mark. In another tube, place 88 cc. H_2O, 5 cc. of H_2SO_4 (sp. gr. 1.84) and cool. Add 5 cc. of 3-per-cent H_2O_2 and then from a burette add the standard titanium solution until the colors match. The volume of the standard solution required multiplied by 100 times its titre represents the percentage of titanium in the sample.

NOTES.

The filtrate and washings from the Silicon Determination should ordinarily be colorless. If light yellow owing to iron, the standard comparison solution should be brought to the same tint with ferric sulfate solution before adding the hydrogen peroxide.

In case the amount of titanium is high the solution may be diluted to a definite volume and aliquot portions taken for the colorimetric test.

DETERMINATION OF IRON

SOLUTIONS REQUIRED

"*Acid Mixture.*"—1200 cc. of H_2SO_4 (25-per-cent by volume), 600 cc. of HCl (sp. gr. 1.20) and 200 cc. of HNO_3 (sp. gr. 1.42)

Tartaric Acid Solution.—Dissolve 20 g. of tartaric acid crystals in 100 cc. of distilled water.

Dilute Ammonium Sulfide Solution.—Add 15 cc. of NH_4OH (sp. gr. 0.90) to 15 cc. of distilled water and saturate with H_2S; then dilute to 200 cc. with distilled water. Use freshly made.

Dilute Sulfuric Acid Solution for Reductor (5-per-cent).—Mix 50 cc. of H_2SO_4 (sp. gr. 1.84) and 1000 cc. of distilled water.

Standard Potassium Permanganate Solution.—Dissolve 1 g. $KMnO_4$ in 1000 cc. of H_2O, and allow to stand for several days in a glass-stoppered bottle in a dark closet. Filter the solution through prepared asbestos and standardize against 0.1000-g. portions of pure sodium oxalate. Each cubic centimeter is equivalent to approximately 0.00177 g. of iron.

METHOD

Dissolve 1 g. of the well-mixed sample in 35 cc. of the acid mixture and proceed as in "Determination of Silicon," including treatment with HF omitting the weighing of residues. Fuse the last residue obtained with a small amount of $K_2S_2O_7$, take up in a little 5-per-cent H_2SO_4, and add to the filtrates and washings obtained. Saturate this solution with H_2S and filter. To the filtrate add 25 cc. of the tartaric acid solution, and then with constant stirring, add NH_4OH (sp. gr. 0.90), finally drop by drop, until it is in slight excess. Pass in H_2S for a few moments, warm somewhat and allow the solution to stand. Filter and wash the sulfide precipitate thoroughly with a freshly made dilute ammonium sulfide solution, until all tartaric acid is washed out.

Dissolve the sulfide precipitate through the filter paper with warm dilute H_2SO_4 (1:10) washing the paper thoroughly, alternately with hot distilled water and with 5-per-cent H_2SO_4. Boil until all H_2S is expelled and add HNO_3 (sp. gr. 1.42) until the iron is oxidized, which is shown by the solution becoming of a yellowish color. Add 3 to 4 g. of NH_4Cl and then NH_4OH (sp. gr. 0.90) in excess until a permanent precipitate forms. Boil again, allow to settle and filter, washing with dilute ammonia and hot distilled water.

Dissolve the washed precipitate through the filter with warm dilute H_2SO_4 (1:10), wash the paper thoroughly with 5-per-cent H_2SO_4 and hot distilled water, using in all about 100 cc. Pass the solution through the reductor and wash the beaker and reductor with 150 cc. of dilute H_2SO_4 for reductor and then 100 cc. of distilled water. Titrate with standard $KMnO_4$ solution.

NOTES

Results of titration should be checked with a blank determination made on corresponding amounts of solutions used.

A small quantity of liquid should always be left in the reductor funnel and air should never be allowed to enter the body of the reductor.

For further details of the use of reductor see "The Chemical Analysis of Iron," by A. S. Blair, or "Quantitive Chemical Analysis," by H. P. Talbot.

The effect of tartaric acid is to form complexes with aluminum, vanadium, chromium, titanium, etc., preventing their precipitation as hydroxides.

Where the iron exists in very small quantities, a gravimetric method is preferable. In this case, double precipitation of iron sulfide is necessary to eliminate all aluminum. Dissolve the second sulfide precipitate in warm dilute H_2SO_4 (1:10), boil, and oxidize the iron with H_2O_2. Add 3 to 4 g. of NH_4Cl and then NH_4OH (sp. gr. 0.90) in considerable excess, with stirring. Filter quickly, dry and ignite the precipitate, weighing as ferric oxide. If an accurate determination of iron is desired, it is necessary to correct for the small accumulation of silica by treatment of the ignited precipitate with H_2SO_4. HF.

DETERMINATION OF COPPER BY ELECTROLYTIC METHOD

APPARATUS FOR ELECTROLYSIS

See the "Determination of Copper by the Electrolytic Method" in the Standard Methods of Chemical Analysis of Manganese Bronze (Serial Designation : B 27) of the American Society for Testing Materials.[1]

SOLUTIONS REQUIRED

Sodium Hydroxide Solution.—Dissolve 250 g. of NaOH in distilled water and dilute to 1000 cc.

Nitric Acid (1:1)—Pour 500 cc. of HNO_3 (sp. gr. 1.42) into distilled water and dilute to 1000 cc.

METHOD

Dissolve 10 g. of the sample in a large casserole or beaker with 150 cc. of the NaOH solution. This should be added in small portions as the action is vigorous for a time. After the action slackens, complete solution may be hastened by warming. When no evidence of further reaction can be seen, dilute to about 700 cc. with water at the boiling temperature. Filter through a strong paper and wash.

Dissolve the residue from the paper with 25 cc. of hot HNO_3 (1:1) into a beaker for electrolyzing, and wash the filter thoroughly with hot water. Boil to expel oxides of nitrogen, add 6 to 8 cc. of H_2SO_4 (1:1), dilute to about 150 cc. and electrolyze. Use a weighed sand-blasted cathode and a current of from 3 to 5 amperes at approximately 10 volts. When the solution is colorless and 1 cc. of the solution gives no copper test with H_2S water on a porcelain plate, remove the solution from the electrodes quickly without interrupting the current. Quickly rinse the cathode in distilled water and then dip it in two successive baths of alcohol. Shake off the excess alcohol and ignite the remainder by bringing it to the flame of an alcohol lamp. Keep the cathode moving continually while the alcohol burns. Weigh as metallic copper.

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[1] 1924 Book of A.S.T.M. Standards.

NOTES

The electrolytic method is preferred for the determination of copper no matter what the percentage.

It is not anticipated that the aluminum will contain tin. In case tin has been found it will be preferable to precipitate with hydrogen sulfide. The sulfides are then to be dissolved, digested in HNO_3 (1 : 1), the metastannic acid filtered off and the electrolysis then carried out.

A staining of the anode indicates the presence of lead or manganese.

Copper, when present in small amounts, may also be determined by potassium cyanide titration. See Standard Methods of Chemical Analysis of Alloys of Lead, Tin, Antimony and Copper (Serial Designation: B 18) of the American Society for Testing Materials.[1]

DETERMINATION OF MANGANESE.

(a) *Persulfate Method.* (For samples containing under 1.5 per cent of Manganese.)

SOLUTIONS REQUIRED.

"Acid Mixture."—Pour 400 cc. of H_2SO_4 (sp. gr. 1.84) into distilled water, cool and add 400 cc. of HNO_3 (sp. gr. 1.42). Then dilute the mixture with distilled water to a total volume of 2000 cc.

Silver Nitrate.—Dissolve 1.33 g. of $AgNO_3$ in 1000 cc. of distilled water.

Stock Sodium Arsenite Solution.—To 15 g. of arsenious oxide (As_2O_3) in a 300-cc. Erlenmeyer flask, add 45 g. of Na_2CO_3 and 150 cc. of distilled water. Heat the flask and contents on the steam bath until the As_2O_3 is dissolved. Cool the solution, filter and make up to 1000 cc. with distilled water.

Standard Sodium Arsenite Solution.—Mix 200 cc. of the stock sodium arsenite solution with 2500 cc. of distilled water, and standardize by the method described below against a steel or iron of known manganese content. One cubic centimeter of this solution should be equivalent to approximately 0.00050 g. of manganese.

Ammonium Persulfate.—Dissolve 6 g. of ammonium persulfate in 100 cc. of distilled water. (This solution should be made up as needed as it deteriorates rapidly.)

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[1] 1924 Book of A.S.T.M. Standards.

METHOD.

In a 250-cc. Erlenmeyer flask, dissolve 1 g. of the sample in 30 cc. of acid mixture by warming on a water bath until solution is complete. Boil until oxides of nitrogen are expelled. Dilute to 100 cc. with boiling hot water, remove the flask from the flame, add 20 cc. of $AgNO_3$ solution, and then 30 cc. of the persulfate solution. Let the solution stand on the steam bath until it has developed a full permanganate color and no bubbles can be seen to come off when the flask is given a whirling motion. Cool to below 25° C., and titrate with the standard arsenite solution to the disappearance of the pink color.

NOTES.

Large amounts (above 0.01 g. of manganese per 100 cc. of solution) of permanganic acid are unstable; the method can, however, be used with satisfaction for alloys containing high percentages of manganese by decreasing the size of the sample.

The ammonium persulfate must be tested for its strength. A good grade should contain about 95 per cent of $(NH_4)_2 S_2O_8$; some lots contain no more than 25 per cent of the salt.

If it is desired, larger amounts of the sample can be used and correspondingly greater amounts of $AgNO_3$, NaCl and $(NH_4)_2S_2O_8$ (preferably in the form of stronger solutions).

(*b*) *Bismuthate Method.* (For samples containing less than 1.50 per cent manganese.) (Optional.)

SOLUTIONS REQUIRED.

Dilute Sulfuric Acid (1 : 1).—Pour 500 cc. of H_2SO_4 (sp. gr. 1.84) into distilled water, cool and dilute to 1000 cc.

Nitric Acid (1 : 3).—Pour 250 cc. of HNO_3 (sp. gr. 1.42) into distilled water and dilute to 1000 cc.

Dilute Nitric Acid (3-per-cent).—Pour 30 cc. of HNO_3 (sp. gr. 1.42) into distilled water and dilute to 1000 cc.

Standard Permanganate Solution.—Dissolve 1 g. of $KMnO_4$ in 1000 cc. of distilled water. Allow it to stand for at least one week and then filter through purified asbestos. Standardize against 0.1 g. portions of pure sodium oxalate. One cubic centimeter of this solution should be equal to approximately 0.00035 g. of manganese.

Standard Ferrous Ammonium Sulfate Solution.—Dissolve 12.4 g. of ferrous ammonium sulfate crystals in 950 cc. distilled water, and add 50 cc. of H_2SO_4 (sp. gr. 1.84).

METHOD.

In a 250-cc. Erlenmeyer flask dissolve 0.5 g. of the sample in 10 cc. of H_2SO_4 (1 : 1), adding a drop or two of HNO_3 (sp. gr. 1.42) occasionally.

When solution is complete heat to expel all oxides of nitrogen and cool. Dilute with 50 cc. of HNO_3 (1 : 3) and add 0.5 g. sodium bismuthate. Heat for a few minutes until the purple color has disappeared with or without the precipitation of manganese dioxide. Add a little ferrous ammonium sulfate solution until the solution becomes clear and boil until the oxides of nitrogen are expelled. Cool, add an excess of sodium bismuthate and agitate for a few minutes. Add 50 cc. of 3-per-cent HNO_3 and filter through an alundum crucible or an asbestos pad. Wash with 50 cc. of 3-per-cent HNO_3. Add from a pipette or a burette 10 to 50 cc. (depending on the amount of permanganic acid) of ferrous ammonium sulfate solution and titrate with the $KMnO_4$ solution.

In exactly the same manner carry through a blank determination using the same amounts of H_2SO_4, HNO_3, and sodium bismuthate as was done with the regular sample. Finally add the exact volume of ferrous ammonium sulfate solution employed and titrate with the $KMnO_4$ solution.

The difference between the volumes required in the two titrations represents the manganese in the sample, and the percentage is found by multiplying this volume by 200 times the manganese titre of the permanganate solution.

NOTES.

Large amounts (above 0.01 g. of manganese per 100 cc. of solution) of permanganic acid are unstable; the method can be used with satisfaction for alloys containing high percentages of manganese by decreasing the size of the sample.

The filtrate from the bismuthate must be perfectly clear, as the least particle of bismuthate carried into the filtrate will vitiate the results.

The solution must be cold at time of filtration and titration.

Instead of employing the method of reducing permanganic acid by means of standardized ferrous ammonium sulfate solution and titrating the excess of the reagent, it is possible to reduce the permanganic acid by standard sodium-arsenite solution. See "Determination of Manganese by the Persulfate Method."

The sodium bismuthate reagent should be tested for manganese and used only if its absence is shown.

ANALYSIS OF LIGHT ALUMINUM ALLOYS.

DETERMINATION OF SILICON, TITANIUM AND IRON.

DETERMINATION OF TOTAL SILICON.

Proceed as described under "Determination of Silicon" in Aluminum.

NOTE.

In case lead sulfate is present (as evidenced by a white crystallin precipitate quickly settling after stirring to the bottom of the casserole) the filtered precipitate must be washed free from the sulfate with an $HN_4C_2H_3O_2$ wash solution made up as follows: Take 20 cc. NH_4OH (sp. gr. 0.90) add 20 cc. of distilled water and make slightly acid with glacial acetic acid. The final washing must be made with hot water.

DETERMINATION OF TITANIUM.

(*a*) *In the absence of nickel.*

METHOD.

Proceed as described under "Determination of Titanium" in Aluminum.

NOTE.

Vanadium, which interferes in the test, should not be encountered in this class of material. Its presence would be betrayed by an "off" reddish-brown cast in the peroxidized solution.

(*b*) *In the presence of nickel.*

METHOD.

Dissolve 1 g. of the sample as described under "Determination of Tin and Antimony" below. If the nitric acid solution of the sodium hydroxide insoluble is not clear it must be filtered, the residue burned in platinum, fused with a little $K_2S_2O_7$, dissolved in a little 5-per-cent H_2SO_4 and added to the nitric acid filtrate. Dilute the nitric acid solution to 200 cc., treat with NH_4OH (sp. gr. 0.90) until ammoniacal, boil 3 minutes, filter and wash. Dissolve the precipitate in hot dilute H_2SO_4 (25-per-cent) keeping the volume below 80 cc., cool and proceed with the colorimetric test as described under "Determination of Titanium" in Aluminum.

NOTES.

The above method provides for the color interference of copper and nickel when they are present in large amounts.

Vanadium (see Notes above) and other elements which would cause color interferences are not apt to occur in this class of material.

DETERMINATION OF IRON.

SOLUTIONS REQUIRED.

Potassium Permanganate Solution (10-per-cent).—Dissolve 10 g. of $KMnO_4$ in distilled water and dilute to 100 cc.

Dilute Sulfuric Acid for Reductor.—Mix 50 cc. of H_2SO_4 (sp. gr. 1.84) and 1000 cc. of distilled water.

Standard Potassium Permanganate Solution.—Dissolve 1 g. of $KMnO_4$ in 1000 cc. H_2O, and allow to stand for several days in a glass-stoppered bottle in a dark closet. Filter the solution through prepared asbestos and standardize against 0.1000-g. portions of pure sodium oxalate. Each cubic centimeter is equivalent to approximately 0.0018 g. of iron.

METHOD.

See "Determination of Titanium."

Transfer the solution used in the "Determination of Titanium" from the Nessler tube to a 400-cc. beaker, add 10-per-cent $KMnO_4$ solution to a good permanent pink tint and pass the solution through the reductor. Wash the beaker and reductor with 150 cc. of the H_2SO_4 for reduction and then with 100 cc. of distilled water. Titrate with the standard $KMnO_4$ solution.

The results of the titration should be corrected by a "blank" determination made on 100 cc. of a 7-per-cent H_2SO_4 solution which has been carried through as in the titanium and iron determinations, not omitting the treatment with iron-free zinc, hydrogen peroxide, and permanganate preliminary to the actual reduction. About 0.5 cc. of the permanganate solution will be required to give a permanent color to the blank.

Calculate the apparent percentage of iron. To obtain the true percentage, multiply the percentage of titanium by 1.161 and subtract.

NOTES.

For description and further details of use of reductor see "Chemical Analysis of Iron," by A. A. Blair, or "Quantitative Chemical Analysis," by H. P. Talbot.

Air should never be allowed to enter the body of the reductor.

Titanium is reduced to the trivalent condition in the reduction process and afterwards oxidized to the quadrivalent condition by the permanganate. There is a slight tendency toward oxidation in the receiver, but this may be disregarded in the small amounts of titanium ordinarily involved.

In case titanium is present and iron alone is to be determined proceed as follows: Place the H_2SO_4 solution of the sample in a 250-cc. flask, dilute to 100 cc. containing 2.5 cc. H_2SO_4 (sp. gr. 1.84) and treat with H_2S for 30 minutes in the cold and 15 minutes while warmed. If sulfides appear, filter and gas the filtrate 10 minutes more. Add 15 cc. H_2SO_4 (1 : 1) and boil vigorously until H_2S is expelled and the volume reduced to 50 cc. Fill the flask with distilled water, transfer to a beaker, and titrate with $KMnO_4$ solution.

In case the alloy contains high percentages of copper, lead, tin or antimony it is preferable to treat the combined filtrate and washings from the silicon determination with H_2S and filter off and wash the sulfates. The solution should then be boiled free from hydrogen sulfide, treated with 10-per-cent $KMnO_4$ solution to a pink tint and then reduced as directed.

Reducible elements other than iron and titanium must be absent. Such elements are chromium, vanadium, molybdenum, tungsten and uranium, and they ordinarily will not be found in this class of material.

DETERMINATION OF ANTIMONY, TIN, LEAD AND COPPER.

PRELIMINARY SEPARATION.

SOLUTIONS REQUIRED.

Sodium Hydroxide Solution (25-per-cent).—Dissolve 250 g. of NaOH in distilled water and dilute to 1000 cc. If a sediment forms on standing, decant the solution and filter through an asbestos pad. The NaOH must be entirely free from organic matter.

Nitric Acid (1 : 1).—Pour 500 cc. of HNO_3 (sp. gr. 1.42) into distilled water and dilute to 1000 cc.

METHOD.

Dissolve 5 g. of the sample in a covered porcelain dish of suitable size in 125 cc. of (25-per-cent) NaOH solution. When solution is complete dilute with 100 cc. of distilled water, decant off the clear solution and wash the residue well with hot dilute NaOH solution (2-per-cent). Treat the filtrate and washings with H_2S. If no precipitate appears, zinc is absent. In case a precipitate appears, zinc must be determined in a fresh sample as described further. Unfold the wet paper containing the NaOH insoluble and wash the residue back into the original vessel. Replace the paper in the funnel and pour 20 cc. of hot HNO_3 (1 : 1) over the paper and allow it to filter into the original vessel. Boil the residue until action ceases and oxides of nitrogen are expelled. Add 50 cc. of hot distilled water and allow to stand and settle for about one hour, keeping the temperature just below the boiling point. Filter on an ashless paper, being careful to keep the solution hot throughout the process of filtration, and recleving the filtrate in a 200-cc. beaker of the tall type used in electrolysis. Wash with boiling hot water. Retain the filtrate and washings for subsequent use in the copper and lead determinations and treat the residue as described under "Determination of Antimony."

NOTES.

Antimony will be quantitatively recovered with the tin provided it does not exceed one-fifth its amount. In the rare case of high antimony evaporate the nitric acid solution of the NaOH insoluble to dryness, bake at 105° C., take up in hot 10-per-cent by volume nitric acid, and proceed with the filtration and washing.

The metastannic acid precipitate will not contain enough copper to make a recovery worth while in ordinary work. As iron is determined in a separate portion and does not interfere in the determinations of antimony and tin, it may also be disregarded.

DETERMINATION OF ANTIMONY

SOLUTIONS REQUIRED.

Standard Permanganate Solution.—The solution prepared as under "Determination of Iron" may be employed. It must be standardized against weighed 0.05-g. portions of pure antimony or suitable material of known antimony content carried through the regular procedure after solution in acid and treatment in the Kjeldahl flask.

METHOD.

See "Preliminary Separation."

Transfer the filter containing metastannic acid and antimony oxide to a 500-cc. Kjeldahl flask, add 10 to 12 cc. H_2SO_4 (sp. gr. 1.84) and 3 to 5 g. Na_2SO_4. Heat until all organic matter is decomposed. Dilute, when cool, with 20 cc. of water. Add 0.5 g. Na_2SO_3 and boil until all SO_2 is expelled from the flask. Dilute to about 250 cc., add 20 cc. of HCl (sp. gr. 1.20) cool to 10° C. and titrate with the $KMnO_4$ solution to the first definite shade of pink. Reserve the solution for the "Determination of Tin."

DETERMINATION OF TIN.

SOLUTIONS REQUIRED.

Standard Iodine Solution.—Dissolve 10.7 g. of iodine in 50 cc. of water containing 20 g. of KI in solution, and dilute to 1000 cc. *when the iodine is completely in solution.* Standardize against pure tin by the method described below.

Starch Solution.—Prepare as described under "Determination of Copper, Iodide Method."

METHOD.

See "Determination of Antimony."

Transfer the solution used in "Determination of Antimony" to a 450-cc. Erlenmeyer flask, add an excess of powdered antimony and 50 cc. of HCl (sp. gr. 1.20). Attach a reflux condenser and boil for 15 minutes on a hot plate. Cool in a current of CO_2, add 5 cc. of starch solution, and without disconnecting the stream of CO_2, titrate with the iodine solution to the blue end point.

DETERMINATION OF COPPER AND LEAD SIMULTANEOUSLY BY THE ELECTROLYTIC METHOD.

APPARATUS REQUIRED.

See "Determination of Copper" under "Analysis of Aluminum."

METHOD.

See "Preliminary Separation."

Dilute the filtrate and washings from the "Preliminary Separation" to about 150 cc. with distilled water and electrolyze, using weighed sand-blasted cathode and anode. Cover with a pair of split watch glasses and use a current of from 1.25 to 1.5 amperes, at approximately 10 volts for each solution. After about one hour the lead will have been entirely deposited on the anode as PbO_2; without interrupting the current add to the electrolyte 3 to 4 cc. of H_2SO_4 (sp. gr. 1.84), wash down the cover glasses, electrodes and sides of the beaker and continue the electrolysis for 15 minutes. If no darkening of the newly-exposed anode surface can be detected and if a few drops of H_2S water added to 1 cc. of the electrolyte on a porcelain plate gives no coloration the electrolysis is complete. In this case, remove the solution from the electrodes quickly without interrupting the current. Rinse the electrodes in quick succession with distilled water and then twice with alcohol. Reserve the electrolyte and water washings for the nickel and magnesium determinations. Dry the anode at 210° C. for thirty minutes. Weigh as PbO_2, using the factor for lead 0.8643 instead of the theoretical value. Shake off the excess alcohol from the cathode and ignite the remainder by bringing it to the flame of an alcohol lamp. Keep the cathode moving continually while the alcohol burns. Weigh as metallic copper.

NOTE.

The electrolyte and water washings may be diluted to 500 cc. and one 250-cc. portion taken for the "Determination of Nickel" and the other for the "Determination of Magnesium" as directed further. If the alloy contains copper and no tin, antimony or lead the electrolytic method given under "Determination of Copper" in Aluminum may be employed.

DETERMINATION OF LEAD AS SULFATE.

(OPTIONAL.)

SOLUTIONS REQUIRED.

"*Lead Acid.*"—Mix 300 cc. of H_2SO_4 (sp. gr. 1.84) and 1800 cc. of distilled water. Dissolve 1 g. of lead acetate C.P. in 300 cc. of distilled water and add this to the hot solution, stirring meanwhile. Let stand at least 24 hours and siphon through a thick asbestos filter.

Dilute Alcohol for Washing.—Mix equal parts of denatured alcohol and distilled water.

METHOD.

See "Preliminary Separation."

To the combined filtrate and washings from the "Preliminary Separation" add 120 cc. of lead acid, and evaporate until copious fumes of H_2SO_4 are evolved. Cool, add 105 cc. of distilled water to dissolve the salts and to make the concentration the same as in the lead acid, heat to boiling, and allow to cool and settle for 5 hours, or over night if convenient. Filter on a weighed porcelain Gooch crucible and wash with lead acid. Set the filtrate and washings aside for the "Determination of Copper by the Iodide Method." Continue the washing and wash out the lead acid with dilute alcohol. Set the Gooch crucible inside a porcelain crucible, dry and ignite for 5 minutes at the full heat of a Tirrill burner. Cool and weigh as $PbSO_4$, which contains 68.32 per cent lead.

DETERMINATION OF NICKEL BY THE DIMETHYLGLYOXIME METHOD.

SOLUTIONS REQUIRED.

Sulfuric Acid (1 : 1).—Pour 500 cc. of H_2SO_4 (sp. gr. 1.84) into distilled water, cool, and dilute to 1000 cc.

Hydrogen Sulfide Wash Water.—Add 10 cc. of H_2SO_4 (sp. gr. 1.84) to 1000 cc. of distilled water and saturate with H_2S.

Tartaric Acid Solution (20-per-cent).—Dissolve 20 g. of tartaric acid in distilled water, dilute to 100 cc. and filter if necessary.

Dimethylglyoxime Solution (1-per-cent).—Dissolve 10 g. of the reagent in 1000 cc. of 95-per-cent alcohol.

METHOD.

Dissolve 1 g. of the sample in 25 cc. of H_2SO_4 (1 : 1) as directed in "Determination of Manganese by the Persulfate Method." After oxides of nitrogen have been expelled add 14 cc. NH_4OH in order to bring down the acidity to approximately 5 cc. H_2SO_4 (sp. gr. 1.84) per 100 cc. of solution. Treat with hydrogen sulfide. Filter off the sulfides, wash with hydrogen sulfide wash water and boil the filtrate until H_2S is expelled. Add a few crystals of $(NH_4)_2S_2O_8$ and boil thoroughly to destroy sulfur and oxidize iron. Add 20 cc. of tartaric acid solution and make slightly ammoniacal. If no precipitate appears add HCl (sp. gr. 1.20) to slight acidity. If a precipitate appears, dissolve in HCl add 10 cc. of tartaric acid and again make ammoniacal, repeating the operation, if necessary, to get a clear solution. To the weakly acid solution add dimethylglyoxime in such an amount that the ratio of the reagent to nickel is at least 4 : 1. Heat the solution to boiling, and make slightly ammoniacal. Allow to digest while cooling for two hours. Collect on a weighed Gooch crucible and wash thoroughly with hot water. Dry for forty-five minutes at 110 to 120° C. and weigh. The nickel dimethylglyoxime contains 20.32 per cent of nickel.

NOTES.

In case copper and lead have been determined electrolytically, nickel may be determined in one 250-cc. portion of the electrolyte and washings (see Notes), starting with the $(NH_4)_2S_2O_8$ treatment to oxidize iron and then proceeding as directed in the method above.

Nickel may be precipitated by glyoxime, filtered, dissolved in nitric acid, made ammoniacal and titrated with cyanide solution according to Frevert's method.[1]

DETERMINATION OF MAGNESIUM.

SOLUTIONS REQUIRED.

Dilute Hydrochloric Acid (1 : 1).—Pour 500 cc. of HCl (sp. gr. 1.20) into distilled water and dilute to 1000 cc.

Sulfuric Acid Wash Water.—Saturate 500 cc. of a 1-per-cent H_2SO_4 solution with H_2S.

Ammonium Sulfide Wash.—Take 10 cc. of NH_4OH, dilute to 500 cc., add 10 g. NH_4Cl and saturate with H_2S.

Microcosmic Salt Solution.—Prepare a clear saturated solution as required.

Ammoniacal Wash Solution.—Dissolve 50 g. of NH_4NO_3 in distilled water, add 50 cc. NH_4OH (sp. gr. 0.90) dilute to 1000 cc. and filter if necessary.

METHOD.

Dissolve 10 g. of the sample and wash the residue as directed under the "Preliminary Separation Determination of Antimony, Tin, Lead and Copper." Dissolve the residue in 10 cc. hot HCl (1 : 1) using a little HNO_3 if necessary. If the hydrogen sulfide group of metals are present, neutralize with NH_4OH, make weakly acid with HCl, dilute to 100 cc., heat and precipitate with hydrogen sulfide. Filter off the sulfides and wash with the H_2SO_4 wash water. To this filtrate or to the HCl solution if no H_2S treatment was required, add 5 cc. HCl (sp. gr. 1.20) make slightly ammoniacal and precipitate with H_2S. Let the solution stand for about two hours at a temperature of approximately 40° C. (at the side of the steam bath). Filter and wash with the $(NH_4)_2S$ wash. Acidify the filtrate with HCl, boil off H_2S, add $(NH_4)_2S_2O_8$ and boil to destroy sulfur if necessary. Add 1 g. of tartaric acid and 30 cc. of microcosmic salt solution. Cool, and add NH_4OH (sp. gr. 0.90) drop by drop while stirring vigorously until a crystallin precipitate begins to form. Continue the very slow addition of NH_4OH with stirring until a precipitate no longer forms and then add one-tenth the volume of NH_4OH (sp. gr. 0.90) and let stand over night. Filter and wash with cold ammoniacal wash solution. Ignite slowly under oxidizing conditions in a tared porcelain or platinum crucible and finally heat over the blast lamp or its equivalent. Weigh as $Mg_2P_2O_7$. The weight multiplied by 0.2185 represents magnesium.

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[1] A. A. Blair, "The Chemical Analysis of Iron," 8th Edition, Lippincott and Co., pp. 182–183.

NOTE.

In case copper and lead were determined electrolytically one reserved portion of the electrolyte and washings (see Notes) may be used for the determination of magnesium, starting in with the addition of 5 cc. HCl (sp. gr. 1.20) and the precipitation of the $(NH_4)_2S$ group.

DETERMINATION OF MANGANESE.

(a) *Persulfate or Bismuthate Method.* (For samples containing under 1.50 per cent manganese.)

Proceed as directed in "Determination of Manganese" under "Analysis of Aluminum."

(b) *Nitric Acid and Potassium Chlorate Method.*[1] (For samples containing over 1.50 per cent manganese.)

SOLUTIONS REQUIRED.

Hydrochloric Acid (1 : 1).—Mix 500 cc. of HCl (sp. gr. 1.20) and 500 cc. of distilled water.

Sulfurous Acid.—Pass SO_2 from a cylinder into 500 cc. of cold distilled water until saturated.

Bromine Water.—Place 20 to 30 cc. of liquid bromine in a 500-cc. bottle, fill with cold distilled water, and shake thoroughly. *Avoid any contact with the skin.*

Dilute Hydrochloric Acid (1 : 3.)—Pour 250 cc. of HCl (sp. gr. 1.20) into distilled water and dilute to 1000 cc.

Sodium-Ammonium Phosphate Solution.—Prepare this solution as needed by dissolving 1 part of the salt in 6 parts of cold water and filtering.

Ammonium Nitrate Solution.—Dissolve 100 g. of NH_4NO_3 in distilled water, make slightly ammoniacal, dilute to 1000 cc. and filter if necessary.

METHOD.

In a 250-cc. Erlenmeyer flask dissolve 1 g. of the sample in 30 cc. of. HCl (1 : 1). When solution is complete add 25 cc. HNO_3 (sp. gr. 1.42) and evaporate until the solution is almost syrupy. Add 50 cc. of HNO_3 (sp. gr. 1.42) and 5 g. $KClO_3$. Heat the solution to boiling either on the hot plate or on a tripod with a thin piece of sheet asbestos, about 1 in. (25 mm.) in diameter, on the center of the wire gauze.

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[1] A. A. Blair, "The Chemical Analysis of Iron," 8th Edition, J. B. Lippincott and Co., pp. 108–115.

Boil the solution 15 minutes, remove from the heat, add 50 cc. HNO_3 (sp. gr. 1.42) and 5 g. $KClO_3$ and boil 15 minutes longer or until yellowish fumes are no longer given off. Cool the solution rapidly in cold water and filter through asbestos. Wash two or three times with HNO_3 (sp. gr. 1.42) (free from oxides of nitrogen which dissolve the dioxide).

Transfer the dioxide with the asbestos filter to the beaker in which the precipitation was made. Pour from 10 to 40 cc. of strong sulfurous acid through the filtering apparatus into the beaker. As soon as the precipitate has dissolved, filter from the asbestos into a 150-cc. beaker, wash with hot water, and add 5 cc. HCl (sp. gr. 1.20). Heat to expulsion of SO_2, add bromine water until the solution is strongly colored with it, and boil off all excess bromine. Add NH_4OH in slight excess, boil for a few minutes, and filter into a 600-cc. beaker. Wash with hot water, remove and reserve the filtrate, and dissolve the precipitate in dilute HCl (1 : 3), allowing the solution to run back into the beaker in which the precipitation was made. Wash the filter with hot water. Boil the solution for 10 minutes to drive off any chlorine and reprecipitate by NH_4OH as before, filter into the reserved filtrate and repeat the solution, precipitation and filtration, allowing all the filtrates from the $Fe(OH)_3$ to run into the 600-cc. beaker. Acidulate this solution, which will be about 300 or 400 cc. in volume, with acetic acid, heat to boiling and treat with H_2S for 10 minutes. Filter, wash with a little dilute acetic acid saturated with hydrogen sulfide and boil off the hydrogen sulfide after adding 5 cc. of HCl (sp. gr. 1.20). Add from 5 to 20 cc. of a clear filtered solution of microcosmic salt, heat to boiling, and add NH_4OH drop by drop with constant stirring. When the precipitate begins to form, stop the addition of NH_4OH and stir until the precipitate becomes crystallin. When this change occurs add one more drop of ammonia; the additional precipitate formed will be curdy, but a few seconds stirring will change it to the silky crystallin condition. Continue the addition of NH_4OH in exactly the same manner until the precipitate is all down and further additions of NH_4OH fail to change the silky appearance. Add a dozen drops of NH_4OH in excess and cool the beaker in ice water. Filter the precipitate on an asbestos filter, wash with the cold NH_4NO_3 solution until the filtrate gives no reaction for HCl. Dry, ignite and weigh as $Mn_2P_2O_7$ which contains 38.69 per cent manganese.

NOTES.

The Ford method is recommended only where extreme accuracy is desired, otherwise use the persulfate or bismuthate methods.

If a cylinder of SO_2 is not available, hydrogen peroxide will serve equally well for the solution of the hydrated manganese dioxide.

DETERMINATION OF ZINC.

(*a*) *Samples containing up to 3 per cent of zinc.*

SOLUTIONS REQUIRED.

Dilute Hydrochloric Acid (1 : 1).—Pour 500 cc. of HCl (sp. gr. 1.20) into distilled water and dilute to 1000 cc.

Acid Wash Water.—Pour 10 cc. of H_2SO_4 (sp. gr. 1.84) into 1000 cc. of distilled water and saturate with H_2S.

Formic Acid Mixture.—Treat 400 cc. of formic acid with 60 cc. of NH_4OH (sp. gr. 0.90) and dilute to 2000 cc.

Formic Acid Wash Water.—Pour 25 cc. of formic acid mixture into 1000 cc. of distilled water and saturate with H_2S.

Dilute Hydrochloric Acid (1 : 10).—Pour 100 cc. of HCl (sp. gr. 1.20) into distilled water and dilute to 1000 cc.

Bromine Water.—See "Determination of Manganese, Nitric Acid and Potassium Chlorate Method."

METHOD.

Dissolve 1 g. of the sample in a covered 400-cc. beaker in 25 cc. of dilute HCl (1 : 1), with the addition of 1 cc. of HNO_3 (sp. gr. 1.42) toward the end of the reaction. Boil to expel oxides of nitrogen and dilute to 200 cc. Precipitate the H_2S group, filter and wash with acid wash water. Boil thoroughly to expel H_2S, cool, and add 5 g. of citric acid crystals. Neutralize with NH_4OH, using methyl orange as an indicator and then add 25 cc. of the formic acid mixture. Dilute to 300 cc., heat nearly to boiling and pass in H_2S until the solution is cold. Filter and wash with the formic acid wash water. Dissolve the zinc sulfide off the filter with warm dilute HCl (1 : 10) and wash the filter with hot water. Boil the solution to expel H_2S. Cool, add 1 g. of citric acid crystals, neutralize with NH_4OH, using methyl orange as an indicator and then add 20 cc. of the formic acid mixture. Dilute to 200 cc. and proceed as before through the filtration on an ashless paper and washing of the sulfide. Dissolve the precipitate with hot dilute HCl, and transfer the solution to a weighed platinum or porcelain dish or crucible. Add a few drops of H_2SO_4 (sp. gr. 1.84) and evaporate the solution until copious fumes escape. If the solution is not clear and colorless, cool, add a few cubic centimeters of HNO_3 (sp. gr. 1.42) and again evaporate the solution until fumes of H_2SO_4 come off freely. Repeat the treatment with HNO_3 if necessary, until the organic matter is destroyed, and the solution is colorless. Remove the excess of H_2SO_4 by heating the dish cautiously, and finally heat at a temperature just below dull redness. Weigh as $ZnSO_4$, which contains 40.49 per cent of zinc.

NOTE.

The use of a sodium hydroxide solution of the turnings is not recommended for the determination of zinc on account of uncertainty as to the completeness of the solution of the zinc in all cases.

(*b*) *Samples containing over 3 per cent of zinc.*[1]

SOLUTIONS REQUIRED.

Ferrous Sulfate Solution.—Dissolve 3 g. of $FeSO_4 . 7H_2O$ in distilled water, add 10 cc. of HCl (sp. gr. 1.20) and dilute to 1000 cc.

Ferrocyanide Solution.—Dissolve 44 g. of K_4FeCN_6 and 0.3 g. of K_3FeCN_6 in distilled water, dilute to 1000 cc. and age six weeks before standardization as follows: Dissolve 6 g. of zinc of known content in 40 cc. of HCl (sp. gr. 1.20) in a 2-liter flask. Cool and make up to the mark. Take 100-cc. portions, add 20 cc. of HCl (1 : 1) and add 13 cc. of NH_4OH (sp. gr. 0.90) and proceed with the titration as directed below.

METHOD.

Proceed as in the previous method until the first zinc sulfide precipitate has been obtained. Return the paper and precipitate to the original beaker and add 20 cc. of HCl (1 : 1). When the sulfide has dissolved, filter off the paper together with some sulfur and possibly a little nickel sulfide. Boil the solution containing the zinc to expel H_2S, cool, and wash down. Add 13 cc. of NH_4OH (sp. gr. 0.90) and if the solution is not alkaline, add NH_4OH carefully until it is. Make the solution barely acid again with HCl (1 : 1) and add 3 cc. of HCl (sp. gr. 1.20) in excess. Add 1 cc. of $FeSO_4$ solution, dilute nearly to 200 cc., heat almost to boiling and titrate with the K_4FeCN_6 solution. The end point is a sharp change in the color of the solution from a turquoise blue to a "pea green" and with several more drops to a "creamy yellow."

NOTE.

The end point occurs a little sooner than the one with uranium nitrate, and is easier to use, as the change is seen directly in the solution.

DETERMINATION OF SODIUM.

SOLUTIONS REQUIRED.

Dilute Nitric Acid (1 : 2).—Pour 250 cc. of HNO_3 (sp. gr. 1.42) into distilled water and dilute to 750 cc.

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[1] Method of F. G. Breyer, 8th International Congress of Applied Chemistry, 1, 162; see also "Analysis of Copper," G. L. Heath, McGraw-Hill Book Co., pp. 250, 251.

Saturated Ammonium Carbonate Solution.—Small quantities made up as required.

METHOD.

Dissolve 5 g. of the sample in a porcelain dish in 100 cc. of dilute HNO_3 (1 : 2) by continued digestion at 50 to 60° C. When solution is complete, evaporate the solution to dryness and heat on a sand bath for several hours to complete decomposition of aluminum nitrate, but without melting any sodium nitrate formed. Cool the residue, take it up in boiling distilled water, filter and wash thoroughly. Treat the water extract with saturated $(NH_4)_2CO_3$ solution, heat to boiling and filter if a precipitate forms. Evaporate the clear filtrate in a weighed platinum dish, treat the residue with sufficient H_2SO_4 to convert the nitrate to sulfate, continue the evaporation and finally heat at 300 to 400° C. to complete expulsion of H_2SO_4. Weigh as Na_2SO_4. A blank should be carried through all operations and the proper correction applied.

DETERMINATION OF NITROGEN.

SOLUTIONS REQUIRED.

Sodium Hydroxide Solution (10-per-cent).—Dissolve 100 g. of NaOH in distilled water, dilute to 1000 cc. and filter through asbestos if the solution develops a sediment upon standing.

Approximate N/10 Sulfuric Acid Solution.—Pour 2.8 cc. of H_2SO_4 (sp. gr. 1.84) into 1000 cc. of distilled water and standardize by any standard method.

Approximate N/10 Sodium Hydroxide Solution.—Dissolve 4.5 g. of NaOH in distilled water and dilute to 1000 cc. This solution should be standardized against the standard acid solution.

METHOD.

Place 3 to 4 g. of the sample in a Kjeldahl flask fitted with a two-hole stopper carrying a separatory funnel and a trap connected with a condenser as in a Kjeldahl determination of nitrogen. Add 400 cc. of the NaOH solution through the separatory funnel, and allow the generated gas to bubble through 50 cc. of N/10 sulfuric acid solution. When the reaction has ceased, boil the alkaline solution to expel all ammonia. Finally titrate the acid with N/10 sodium hydroxide in the usual manner and calculate nitrogen.

DETERMINATION OF ALUMINUM AND ALUMINUM OXIDE.

SOLUTIONS REQUIRED.

Sodium Hydroxide Solution (10-per-cent).—Dissolve 100 g. of NaOH in distilled water and dilute to 1000 cc. If a sediment forms on standing, decant the solution and filter through an asbestos pad.

Sodium Carbonate Solution.—Dissolve 36 g. of Na_2CO_3 in distilled water, dilute to 1000 cc., and filter if necessary.

Barium Chloride Solution.—Dissolve 90 g. of $BaCl_2 . 2H_2O$ in distilled water, dilute to 1000 cc., and filter if necessary.

Dilute Hydrochloric Acid.—Dilute 500 cc. of HCl (sp. gr. 1.20) with 500 cc. of water.

Methyl Red Indicator.—Dissolve 0.02 g. of the acid in 100 cc. of distilled water.

Ammonium Chloride Wash Solution.—Dissolve 50 g. of NH_4Cl in distilled water, make sure the solution is just alkaline to methyl red, dilute to 1000 cc. and filter if necessary.

Dilute Nitric Acid (1 : 1).—Dilute 500 cc. of HNO_3 (sp. gr. 1.42) with 500 cc. of distilled water.

Acid Wash Water.—Add 5 cc. of H_2SO_4 (sp. gr. 1.84) to 500 cc. of water and saturate with H_2S.

PRELIMINARY TREATMENT.

Place 2 g. of the sample in a suitable porcelain dish, add 60 cc. of the NaOH solution (10-per-cent), cover with a glass, and cool in water if the reaction proceeds too violently. When violent action ceases, wash the cover-glass and sides of the vessel, replace the glass and warm on a sand bath or steam-box until reaction ceases. These operations should be carried on expeditiously. Filter and wash the residue with hot NaOH solution (10-per-cent). Reserve the residue and treat the filtrate and washings as below.

(*a*) *Determination of Aluminum.*

Cool the filtrate and washings to room temperature, dilute to exactly 500 cc. and mix thoroughly. Transfer pipetted 50-cc. portions to 600-cc. beakers, add 10 cc. of HCl (sp.gr. 1. 20), place on water bath and evaporate to dryness. When dry, continue the heating for one hour more. Take up the residue in cold distilled water, transfer to a flask and add a mixture of 50 cc. of the Na_2CO_3 solution and 50 cc. of the $BaCl_2$ solution. Shake thoroughly and allow to settle. Filter, wash with cold water, and then transfer the precipitate to the original flask by piercing the paper and washing down with boiling water, finally with dilute HCl, adding enough to dissolve the precipitate and avoiding mechanical loss. Evaporate the solution to one-third volume, add a few drops of methyl red and then NH_4OH (sp. gr. 0.90) until the indicator just turns yellow. Boil for three minutes, filter and wash with warm 5-per-cent NH_4Cl solution. Bring the precipitate back into the beaker as before, dissolving all $Al(OH)_3$ on the paper and evaporate the solution to dryness. Drench with a little HCl (sp. gr. 1.20) add hot water and warm until salts are in solution. Filter in case a residue is present, reprecipitate $Al(OH)_3$ and wash as above. Place the wet paper and contents in a tared crucible (with cover and preferably of platinum), dry, ignite carefully in an oxidizing atmosphere and finally cover and blast strongly. Weigh as Al_2O_3 and calculate aluminum.

NOTES.

This method is a modification of the method proposed by J. G. Rhodin, Faraday Society Trans., *14*, (1914–19) pp. 135–7 and presupposes complete solubility of aluminum and insolubility of aluminum oxide in a 10-per-cent solution of NaOH.

It is important that the "50-cc." pipette deliver exactly one-tenth of the "500-cc." volume.

Two precipitations with NH_4OH are necessary to eliminate barium and the intervening evaporation to dryness is desirable in case silica is present.

(*b*) *Determination of Aluminum Oxide.*

See "Preliminary Separation."

Transfer the residue insoluble in NaOH solution (10-per-cent) to a small flask and treat the paper with hot dilute HNO_3 (1:1). When solution of the residue is complete, dilute to 100 cc., neutralize with NaOH solution (10-per-cent), add 3 cc. HCl (sp. gr. 1.20) and treat with hydrogen sulfide. Filter, wash the sulfides with acid wash water and boil the filtrate and washings until H_2S is expelled, using a few crystals of $K_2S_2O_8$ if sulfur separates. Pour the hot solution slowly and with stirring into 50 cc. of boiling hot NaOH solution (10-per-cent). Filter and wash with a little hot NaOH solution (10-per-cent).

In case the precipitate is large, dissolve it and reprecipitate. Acidify the NaOH filtrate or filtrates with HCl (sp. gr. 1.20) and precipitate $Al(OH)_3$ as described in (*a*) above. A second precipitation should be carried out in case the precipitate is large, and always in accurate work. Ignite, weigh as Al_2O_3 and report as such.

TENTATIVE SPECIFICATIONS

FOR

QUICKLIME FOR USE IN THE MANUFACTURE OF SULFITE PULP[1]

Serial Designation: C 46 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1924, 1925.

Lime in Manufacture of Sulfite Pulp.

1. Lime is used in the "Milk of Lime" or "Tank System" of sulfite pulp manufacture for making the cooking liquor. The milk of lime is held in solution or suspension in a series of tanks equipped with suitable agitators. The sulfur dioxide is forced or drawn through these tanks successively. In some cases, the tanks are built on top of each other in the form of a tower. The contents of the first tank are drawn off when the liquor has reached a certain strength (3.5 to 6 per cent total SO_2) and the contents of the second and third tanks progress to the first and second tanks respectively. The third tank is again charged with fresh milk of lime. There are other systems of absorption which provide for continuous instead of intermittent operation. The function of the lime is to furnish the base for the formation of the bisulfites of calcium and magnesium.

I. REQUIREMENTS.

Requirements.

2. (*a*) Either calcium or magnesium lime may be used, but owing to the greater solubility and reactivity of magnesium bisulfite, high magnesium lime gives the best results. The ratio of MgO to CaO shall be reasonably constant and the lime shall be moderately free from impurities.

(*b*) The quicklime shall conform to the following requirements as to chemical composition, all percentages to be based on the weight of the sample taken at the point of manufacture:[1]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

	Calcium Lime		Magnesium Lime	
	Maximum	Minimum	Maximum	Minimum
Calcium oxide, per cent	...	92.5	....	55.4
Magnesium oxide, per cent	2.5	....	....	39.6
Oxides of silicon, iron and aluminum, per cent	3.0	....	3.0	....

II. METHODS OF TEST.

Chemical Analysis.

3. The chemical analysis of the lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 - 25 T) of the American Society for Testing Materials.[2]

III. SAMPLING, INSPECTION, REJECTION, ETC.

Sampling, Inspection, Rejection, etc.

The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Tentative Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50 - 24 T) of the American Society for Testing Materials.[3]

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[1] If it is desired to use the non-volatile basis the figures given above should be changed to the following:

	Calcium Lime		Magnesium Lime	
	Maximum	Minimum	Maximum	Minimum
Calcium oxide, per cent	....	94.3	....	56.5
Magnesium oxide, per cent	2.6	....	....	40.4
Oxides of silicon, iron and aluminum, per cent	3.1	....	3.1	....

[2] See p. 618.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 844 (1924); also 1925 Book of A.S.T.M. Tentative Standards, p. 246.

TENTATIVE SPECIFICATIONS
FOR
QUICKLIME FOR USE IN WATER TREATMENT[1]

Serial Designation: C 53 – 25 T

This is a Tentative Standard only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924; REVISED, 1925.

Quicklime for Water Treatment.

1. (*a*) In the treatment of water for public supplies, lime is used alone or with iron sulfate or aluminum sulfate to produce a precipitate which assists in the clarification of the water and in the removal of the bacteria by filtration. An excess of lime is sometimes used to remove part of the hardness of the water.

(*b*) Lime and soda ash are used together for softening water.

(*c*) The lime serves as a chemical reagent in water treatment. The only useful constituent of the lime is the calcium oxide capable of reacting with the other chemicals which are in the water or which are added to it. Inert material, besides reducing the value in proportion to its amount, also makes more sludge to be disposed of for a given amount of chemical action and thus reduces the capacity of the equipment in which it is used.

I. REQUIREMENTS

Requirements.

2. (*a*) The lime shall be substantially free of core, ash, and dirt, and shall be capable of disintegrating, in water, into a suspension of finely divided material.

(*b*) The standard quicklime for use in water treatment shall contain 90 per cent of available lime. All percentages enumerated herein are based on the sample taken at the point of manufacture.

II. METHODS OF TEST

Chemical Analysis.

3. The chemical analysis for available lime shall be made in accordance with the Tentative Methods of Chemical Analysis of

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 - 25 T) of the American Society for Testing Materials.[1]

III. SAMPLING, INSPECTION, REJECTION, ETC.

Sampling, Inspection, Rejection, etc.

4. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Tentative Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50 - 24 T) of the American Society for Testing Materials.[2]

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[1] See p. 618.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 844 (1924); also 1925 Book A.S.T.M. Tentative Standards, p. 246.

TENTATIVE SPECIFICATIONS

FOR

HYDRATED LIME FOR USE IN WATER TREATMENT[1]

Serial Designation: C 54 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924; Revised, 1925.

Hydrated Lime for Water Treatment.

1. (*a*) In the treatment of water for public supplies, lime is used alone or with iron sulfate or aluminum sulfate to produce a precipitate which assists in the clarification of the water and in the removal of bacteria by filtration. An excess of lime is sometimes used to remove part of the hardness of the water.

(*b*) Lime and soda ash are used for softening water.

(*c*) Hydrated lime serves as a chemical reagent in water treatment. The only useful constituent of the hydrated lime is the calcium hydroxide capable of reacting with the other chemicals which are in the water or which are added to it. Inert material, besides reducing the value in proportion to its amount, also makes more sludge to be disposed of for a given amount of chemical action and thus reduces the capacity of the equipment in which it is used.

I. REQUIREMENTS

Requirements.

2. The standard hydrated lime for use in water treatment shall contain 90 per cent of available calcium hydroxide (equivalent to 68.1 per cent of calcium oxide). All percentages enumerated herein are based on the sample taken at the point of manufacture.

II. METHODS OF TEST

Chemical Analysis.

3. The chemical test for available lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone,

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

Quicklime and Hydrated Lime (Serial Designation: C 25 – 25 T) of the American Society for Testing Materials.[1]

III. SAMPLING, INSPECTION, REJECTION, ETC.

Sampling, Inspection, Rejection, etc.

4. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Tentative Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50 – 24 T) of the American Society for Testing Materials.[2]

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[1] See p. 618.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 844 (1924); also 1925 Book A.S.T.M. Tentative Standards, p. 246.

TENTATIVE METHODS OF CHEMICAL ANALYSIS
OF
LIMESTONE, QUICKLIME AND HYDRATED LIME.[1]

Serial Designation: C 25 - 25 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1919; REVISED, 1921, 1922, 1925.

TREATMENT OF SAMPLE

Treatment of Sample.

The sample, taken and prepared in accordance with the requirements of the particular specifications of the Society applicable to the material shall be treated as follows:

Weigh out 0.5 g. of the substance and, if a limestone or hydrated lime, ignite in a covered platinum crucible in an electric muffle (Note 1) for 15 minutes, or longer if the heat is not powerful enough to effect complete decomposition within that time. Transfer to an evaporating dish, preferably of platinum (Note 2) for the sake of celerity in evaporation, mix to a thin slurry with distilled water, add 5 to 10 cc. of HCl (sp. gr. 1.20) and digest with aid of gentle heat and agitation until solution is complete. Solution may be aided and the time shortened by light pressure with the flattened end of a glass rod upon resistant lumps. Then evaporate the solution to dryness, so far as this may be possible on the water bath.

DETERMINATION OF SILICA.

Silica.

When dry or nearly so, place the dish and its contents in an air bath or (covered) on a platinum triangle resting on a hot plate and, if it is a high calcium limestone that is under treatment, heat for one hour at 200° C.; if a high magnesian stone is under treatment the temperature should not exceed 120° C. Drench the cooled mass with HCl (sp. gr. 1.20) and allow to stand for a few minutes. Add an equal volume of water, cover the dish and place on the water bath for

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[1] *Bulletin 700*, U. S. Geological Survey, should be used as a reference book in connection with these methods, especially when very exact and detailed analyses are desired. Blank determinations should be made on all reagents, and corrections applied when called for.

Criticisms of these Tentative Methods are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

10 minutes (Note 3). Separate the silica by filtration on a filter of suitable size. Wash thoroughly with dilute HCl and then wash twice with cold water.

Evaporate the filtrate to dryness. Extract the residue with HCl as before but allowing only a few minutes time. Filter this solution through a second and smaller paper.

Transfer, wet, the papers containing the separated residue to a weighed platinum crucible. Char carefully without allowing the paper to inflame and ignite to constant weight in an electric muffle and weigh (Note 1). Correct the weighed silica for foreign matter by evaporating with 5 cc. of HF and one or two drops of H_2SO_4.[1] Heat the residue for 2 or 3 minutes in an electric muffle and again weigh. The difference between this weight and that previously obtained gives the weight of the silica (Note 4).

DETERMINATION OF TOTAL IRON AND ALUMINUM.

Iron and Alumina.

Fuse the residue from the silica determination using a little Na_2CO_3 (Note 5). Dissolve the cooled melt in HCl and add the solution to the filtrate from the second evaporation for silica Add a few drops of bromine water or HNO_3 and boil the solution until all trace of bromine or chlorine is gone. Then add HCl, if not already present, sufficient to insure a total volume of 10 to 15 cc. of HCl (sp. gr. 1.20).[2] Add a few drops of methyl red solution and after diluting to 200 to 250 cc. heat the liquid to boiling. Neutralize by means of NH_4OH (dilute towards the end) until the color of the liquid changes to a distinct yellow. Boil for one or two minutes, allow to settle, filter and wash the precipitate at once two or three times with hot NH_4Cl (2-per-cent) solution and suck dry.[3]

Set the filtrate aside. Dissolve any precipitate from the paper in hot, dilute HCl, the solution passing into the beaker in which the precipitation was made and wash the paper thoroughly with hot water. Boil the solution to expel any trace of chlorine and again precipitate the hydroxides with NH_4OH as described above. Ignite the precipitate, moist (Note 1), in a weighed platinum crucible, cool and weigh as Al_2O_3 and Fe_2O_3 (Note 6).

DETERMINATION OF TOTAL IRON.

Total Iron.

Fuse the combined iron and aluminum oxides (Note 7) in a platinum crucible at a very low temperature with 3 to 4 g. of $K_2S_2O_7$ or better $Na_2S_2O_7$ (Note 8). Take up the melt with so much dilute H_2SO_4 that there shall be not less than 5 g. absolute acid and enough water to effect solution on heating. Evaporate the solution and eventually heat until acid fumes come off copiously. After cooling and redissolving in water, filter out the small amount of silica, wash, ignite, weigh and correct by HF and H_2SO_4 (Note 9). Add the weight so corrected to the weight of silica previously found and deduct from the gross weight of the oxides of iron and aluminum.

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[1] See *Bulletin 700*, U. S. Geological Survey, p. 103.

[2] See *Bulletin 700*, U. S. Geological Survey, p. 258.

[3] See *Bulletin 700*, U. S. Geological Survey, p. 107.

Reduce the filtrate by zinc (Note 10). Titrate with $KMnO_4$ using a N/20 solution.

DETERMINATION OF ALUMINUM.

Aluminum.

Subtract the calculated weight of Fe_2O_3 obtained by the method described above from the weight of $Fe_2O_3 + Al_2O_3$. Report the remainder as Al_2O_3. In case phosphorus is determined, calculate it to P_2O_5 and deduct also.

DETERMINATION OF TOTAL IRON.

ALTERNATIVE METHOD (Note 7).

Total Iron, Alternative.

Dissolve 2 to 5 g. (depending upon the amount of iron present) of the properly prepared sample in HCl and evaporate rapidly to dryness. Treat the residue with water and HCl. filter off the silica and wash several times with hot water. Precipitate the iron in a boiling solution with fixed alkali, allow to settle, filter and wash free of chlorides, using hot water. Dissolve in dilute H_2SO_4.

Ignite the insoluble matter from the evaporated hydrochloric acid solution in a platinum crucible. Treat with H_2SO_4 and HF and heat until fumes of H_2SO_4 appear. Bring the residue into solution with the addition of a few drops more of H_2SO_4 and combine the solution with that of the bulk of the iron. Reduce this solution by zinc and titrate the iron with $KMnO_4$ in the usual manner.

DETERMINATION OF CALCIUM.

(Note 11.)

(*a*) *Gravimetric Method.*

Calcium, Gravimetric.

Add a few drops of NH_4OH to the combined filtrate from the R_2O_3 precipitate, and bring the solution to boiling. To the boiling solution add 35 cc. of a saturated solution of $(NH_4)_2C_2O_4$, and continue the boiling until the precipitated CaC_2O_4 assumes a granular form; then allow it to stand 20 minutes, or until the precipitate has settled and the supernatent liquid is clear, filter and wash thoroughly with boiling water (Note 12). Place the wet filter and precipitate in a platinum crucible, and burn off the paper over the small flame of a Bunsen burner, or in a muffle. Ignite the residue, dissolve in hot dilute HCl, and make up the volume of the solution to 100 cc. with water. Add NH_4OH in slight excess and boil the liquid. If a small amount of $Al_2(OH)_6$ separates, filter it out, wash with NH_4Cl (2-per-cent solution), ignite, weigh and add this weight to that found in the first determination. Then reprecipitate the lime by $(NH_4)_2C_2O_4$, allow to stand until settled, filter, wash thoroughly with boiling water (Note 12) and weigh as calcium oxide after ignition to constant weight in a covered platinum crucible.

(*b*) *Volumetric Method.* (Note 13.)

Calcium, Volumetric.

Make the filtrate from the iron and alumina precipitate alkaline with NH_4OH, boil, and add 35 cc. of a boiling saturated solution of $(NH_4)_2C_2O_4$. Stir vigorously and allow to stand until the precipitate has settled, filter on an 11-cm. paper, and wash ten times with hot water (Note 14). Transfer the paper and precipitate to the beaker in which the precipitation was made, spreading the paper out against the upper portion of the beaker. Wash the precipitate from the paper with a jet of hot water, fold the paper and leave it adhering to the upper portion of the beaker. Add to the contents of the beaker 50 cc. of dilute H_2SO_4 (1:10), dilute to a volume of 250 cc. with hot water and heat to a temperature of 80 to 90° C. Titrate with the standard $KMnO_4$ solution[1] until the pink end point is obtained. Now drop the folded filter paper (which has been adhering to the side of the beaker) into the liquid; the pink color of the latter will be discharged. Finish the titration by adding $KMnO_4$, a drop at a time, until the pink end point is again obtained. A Gooch crucible may be used instead of filter paper. From the total quantity of standard $KMnO_4$ solution used, calculate the percentage of calcium oxide.

DETERMINATION OF STRONTIUM.

Strontium.

Transfer the weighed oxides obtained in the gravimetric determination of calcium to a small flask of 20-cc. capacity and dissolve in HNO_3. Evaporate to dryness and heat at 150 to 160° C. Treat the thoroughly dried nitrates with as little (rarely over 2 cc.) of a mixture of equal parts of absolute alcohol and ether as may be needed to dissolve the calcium salt, solution being hastened by occasional gentle agitation. After standing over night in the corked flask collect the insoluble matter on the smallest possible filter and wash with more of the above mixture of alcohol and ether. After drying, pass a few cubic centimeters of hot water through the filter, on which may remain a few tenths of a milligram of residue, which does not usually contain any lime or other alkaline earth and whose weight is therefore to be deducted from that of the lime, unless it can be shown that it is derived from the glass of the little flask in which the nitrates of calcium and strontium were evaporated. To the solution of strontium nitrate in a small beaker add a few drops of H_2SO_4 and then its volume of alcohol, whereby the strontium is precipitated as sulfate. After twelve hours, filter on a small filter paper and wash the residue with 50-per-cent alcohol. Ignite at a low temperature, moisten with dilute H_2SO_4, evaporate, again ignite and weigh as strontium sulfate. Test the sulfate spectroscopically as to freedom from calcium and barium.

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[1] See directions for preparing this solution.

Calculate the weight of strontium oxide and deduct from the weight of calcium oxide obtained as above.

DETERMINATION OF MAGNESIUM.

Magnesium.

Acidify the combined filtrates from the calcium precipitates with HCl and concentrate on the water bath to about 150 cc., and heat to boiling. To the boiling solution add 10 cc. of a saturated solution of $Na(NH_4)HPO_4$ and continue the boiling for several minutes. Then cool to room temperature, and add NH_4OH drop by drop with constant stirring until the crystallin ammonium-magnesium orthophosphate begins to form and then in moderate excess, the stirring being continued for several minutes. Allow the liquid to stand in a cool atmosphere for from 12 to 48 hours (Note 15) and filter.

Dissolve the precipitate in hot dilute HCl, make up the solution with water to about 100 cc., add 1 cc. of the saturated solution of $Na(NH_4)HPO_4$ and NH_4OH drop by drop with constant stirring until the precipitate is again formed as above described and the ammonia is present in moderate excess. Then allow it to stand in a cool atmosphere for from 12 to 48 hours (Note 15), filter on paper or a Gooch crucible, wash with dilute NH_4OH containing NH_4NO_3 (Note 16), ignite (Note 1), cool, and weigh as $Mg_2P_2O_7$ (Note 17).

DETERMINATION OF TOTAL VOLATILE MATTER.

(LOSS ON IGNITION.)

Volatile Matter.

Place 1 g. of the properly prepared sample (Note 18) in a weighed platinum crucible, cover with the lid and heat gently for 5 minutes (Note 1), gradually increasing the temperature to the maximum of the muffle, and maintain at this temperature until constant weight is obtained (usually about 15 minutes).

DETERMINATION OF MECHANICAL MOISTURE

(LIMESTONE ONLY.) (Note 19.)

Mechanical Moisture.

Weigh 1 g. of the properly prepared sample in a tared, wide, low-form, flat-bottomed weighing bottle. Heat uncovered in a ventilated drying oven at 120° C. for two hours. Quickly stopper and cool in a desiccator. Lift the stopper momentarily just before weighing and weigh. The use of a similar weighing bottle as a counterpoise carried through all of the operations is a desirable procedure. The loss in weight represents "Mechanical Moisture" or "Hydroscopic Water" at 120° C.

DETERMINATION OF CARBON DIOXIDE.

Carbon Dioxide.

Carbon dioxide is to be determined upon the properly prepared sample according to the method given in U. S. Geological Survey Bulletin No. 700, p. 217. An illustration showing the arrangement of the component parts of the necessary apparatus is shown on p. 218 of that Bulletin.

With limestone, use a 0.5-g. sample; with burned lime, hydrated lime, etc., use a 5-g. sample.

Boil the weighed sample with dilute HCl in a small Erlenmeyer flask attached to an upwardly inclined condenser, whence, after passing through a drying system—calcium chloride, anhydrous copper sulfate to retain hydrogen sulfide from decomposable sulfides and any HCl that may pass over, then calcium chloride again—the carbon dioxide is caught by absorption tubes filled with soda-lime followed by calcium chloride. Of course, arrangement is made for a current of air free from CO_2 with which to sweep out the apparatus before and after the experiment, and for a slow current during its continuance.

The results are very accurate and the determination can be quickly carried out

The manipulations are as follows: Pour hot water upon the powder in the flask fitted with a separatory funnel and delivery tube; attach this to the condenser, and force a current of air free from CO_2 through the whole system, except the weighed absorption tubes, until the original air has been displaced, the observation bulbs (containing H_2SO_4 to show the rate of gas flow) being attached directly to the drying system. Then close the stop-cock in the separatory funnel, half fill the latter with HCl (1:1), replace the rubber stopper of the funnel, insert the absorption tubes between the drying system and the observation bulbs, and allow the acid to flow into the flask, slowly if there is much carbon dioxide, rapidly if there is but little. When effervescence diminishes in the former case, at once in the latter, light the burner under the flask and start the flow of water through the condenser. Keep the flame low, so as to secure steady but quiet ebullition, and do not interrupt the air current although it should be reduced to a slow rate. With much carbon dioxide the rate of absorption is very readily noted by holding the hand to the soda-lime tubes.

which become hot or warm when absorption is taking place. Sufficient time having elapsed (Note 20), extinguish the flame, and increase the air current. When cool, disconnect the soda-lime tubes from the apparatus and allow to stand in the balance case until two weights taken 30 minutes apart agree within 0.5 mg.

The soda lime for use in this determination must be porous, not hard and unabsorptive like that sometimes used for the combustion of nitrogenous organic substances.

DETERMINATION OF SULFURIC ANHYDRIDE.

Sulfuric Anhydride.

Place 2 g. of the properly prepared sample in a small, dry beaker and stir it up with 10 cc. of cold water until all lumps are broken up and the lighter particles are in suspension. Add 15 cc. of dilute HCl (1: 1) and heat until solution is complete. Filter through a small paper and wash the residue thoroughly with hot water. Dilute the filtrate to 250 cc., heat to boiling, and add 10 cc. of a boiling 10-per-cent solution of barium chloride drop by drop with constant stirring. Stir well and allow to stand over night. Filter, wash with boiling water, ignite and weigh as $BaSO_4$.

DETERMINATION OF TOTAL SULFUR.

Total Sulfur.

Digest 2 g. of the properly prepared sample on the hot plate with 25 to 30 cc. of bromine water. Then add 15 cc. of dilute HCl (1:1) and boil until solution is complete and all bromine has been expelled. Then proceed as outlined above under "Determination of Sulfuric Anhydride."

DETERMINATION OF PHOSPHORUS.

Phosphorus

Dissolve 10 g. of the sample in 80 cc. of dilute HNO_3 (1:1), filter, and wash the residue with hot water. Ignite, and fuse the residue with a little Na_2CO_3, cool the melt and add its nitric acid solution to the main filtrate. (Note 21.)

In case the rock is rather argillaceous, mix the powder with half its weight of Na_2CO_3 and blast strongly. Dissolve the cooled melt in HNO_3 and evaporate to dryness. Take up the dry residue with dilute HNO_3 (1: 2), filter and wash the residue with hot water. Ignite the residue in platinum, and evaporate with HF and HNO_3. Finally, evaporate twice with HNO_3 alone to expel all fluorine, dissolve the residue in HNO_3 and add the solution to the main filtrate. (Note 21.)

Oxidize the nitric acid solution by boiling with 10 cc. of 1.5-per-cent $KMnO_4$ solution until manganese dioxide is precipitated. Dissolve the oxide by addition of H_2SO_3 or a sulfite free from phosphorus and boil to expel the oxides of nitrogen and sulfur. Neutralize the HNO_3 with NH_4OH and then add 1 cc. of HNO_3 (sp. gr. 1.42) for every 100 cc. of solution. Bring the solution to a temperature of 40° C., add ammonium molybdate solution[1] and shake for 10 minutes. Allow to stand at a temperature of not over 40° C. for 1 to 12 hours. Filter and wash 10 times with a 1-per-cent KNO_3 solution. Return the precipitate to the precipitating vessel, add a measured excess of N/10 NaOH and 25 cc. of water free from CO_2 and shake or stir until the precipitate is dissolved. Dilute to 100 to 200 cc. with water free from CO_2, add 3 drops of 0.2-per-cent phenolphthalein solution, and discharge the pink color with standard acid. Finish the titration by adding standard alkali until the reappearance of the pink color. The alkali solution should be free from carbonate and be standardized against the Bureau of Standards' standard benzoic acid.[2] The ratio of phosphorus to NaOH should be considered as 1 : 23 in calculating results. (Note 22.)

DETERMINATION OF MANGANESE.

Manganese.

Dissolve 10 g. of the sample in 100 cc. of dilute HNO_3 (1 : 1), filter and wash the residue with hot water. Ignite the residue in platinum, fuse with a little Na_2CO_3 and add the nitric acid solution of the melt to the main filtrate.

Dilute the solution to 150 cc. and add 0.5 g. of sodium bismuthate (Note 23). Heat for a few minutes, or until the pink color has disappeared and dioxide has precipitated. If manganese dioxide does not precipitate add more bismuthate. Clear the solution by adding a few drops of a saturated solution of sodium bisulfite or other suitable reducing agent free from chlorides, and boil to expel all oxides of nitrogen and sulphur. Cool to 15° C., add an excess of sodium bismuthate, agitate and let stand for a few minutes. Add 50 cc. of 3-per-cent HNO_3 and filter through asbestos. Wash with 3-per-cent HNO_3 until the washings run through colorless. Add a measured excess of a standard $FeSO_4$ solution and titrate back with a standard $KMnO_4$ solution of which the strength has been determined by means of the Bureau of Standards sodium oxalate. (Note 24.)

DETERMINATION OF FERROUS IRON SOLUBLE IN SULFURIC ACID.

Ferrous Iron.

In limestones, the presence of carbonaceous matter renders the exact or even approximate determination of ferrous iron often impossible. Nevertheless, even in its presence acceptable results are sometimes obtainable if there is not much of such matter and if it does not give with acid a colored solution. Occasionally limestones show films of manganese peroxide, which likewise interferes with the determination.

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[1] See Blair, "The Chemical Analysis of Iron," 8th Edition, p. 92.

[2] See *Journal*, Am. Chem. Soc., Vol. 34, p. 1027 (1912), and Vol. 35, p. 1309 (1913).

(*a*) *In Absence of Carbonaceous Matter.*

The powder, one to several grams, is introduced into a stout flask of about 200 to 250-cc. capacity and boiled with a little water till all air is expelled. While still boiling, dilute H_2SO_4 is added, a little at a time, till effervescence ceases, and then a further amount. Calcium sulfate precipitates, but the iron will remain in solution. The flame is then removed and a stopper tightly inserted, through which passes a small stop-cock funnel. When cool, or nearly so, cold water is poured into the funnel, the cock cautiously opened, and the water drawn into the flask, more water being poured into the funnel as fast as it empties, till the solution amounts to 100 to 150 cc. Such precaution to exclude air is hardly necessary in most cases, however, for in presence of H_2SO_4 the oxidation of ferrous iron is exceedingly slow. It is ordinarily quite sufficient to equalize the internal and external pressures by opening the cock, removing the stopper, and pouring in cold water. The flask is then brought under a burette containing dilute $KMnO_4$ solution and the iron titrated without delay. With a carbonate which is wholly decomposable without the aid of heat, solution may be accomplished in a flask filled with CO_2, using cold acid, whereby the danger of attack of silicates is lessened if these are present. If the preference is for the $K_2Cr_2O_7$ method of titration, HCl may be used instead of H_2SO_4, in absence of manganese peroxide. In this case there is, of course, no separation of an insoluble calcium salt, a fact which renders easier the subsequent determination of the iron in any insoluble residue the rock may yield. The ferrous iron thus found is mostly, if not altogether, that existing as carbonate.[1]

(*b*) *In Presence of Carbonaceous Matter.*

Decomposition is effected in a flask by dilute H_2SO_4 in an atmosphere of CO_2. With limestones and active agitation no heat need be used, but with dolomites it will be necessary. The solution is then quickly filtered through asbestos (in an atmosphere of CO_2 if much iron is present), the residue and filter washed a few times with water, and the filtrate titrated at once with permanganate. If it is colored by organic matter, the result may be in error. As above, HCl and titration by $K_2Cr_2O_7$ may be used in absence of manganese peroxide. In either case it is important to allow the acid to act no longer than is necessary and to filter quickly. A determination of the ferrous iron in the insoluble matter is usually not worth attempting, because of the admixed organic matter.

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[1] If a determination of ferrous iron in the insoluble residue is desired, see U. S. Geological Survey *Bulletin 700*, p. 265.

DETERMINATION OF AVAILABLE LIME IN QUICKLIME AND HYDRATED LIME BY THE MODIFIED SCAIFE METHOD

Available Lime.

"Available lime" in quicklime or hydrated lime is that (or those) constituent (or constituents) which enters (or enter) into the reaction under the conditions of the specified method or process.

The interpretation of results obtained by the following method shall be restricted by the above definition.

Place 1.4 g. of the carefully prepared and finely ground (passing a No. 100 sieve) lime in a 400-cc. beaker, add 200 cc. of hot water, cover, heat carefully and then boil for three minutes.

Cool, wash down cover, add two drops of phenolphthalein and titrate with normal hydrochloric acid, adding the acid dropwise as rapidly as possible and stirring vigorously to avoid local excess of acid. When the pink color disappears in streaks, retard the rate of addition of acid somewhat, but continue until the pink color disappears entirely and does not reappear for 1 or 2 seconds. Note the reading and ignore the return of color.

Repeat the test, substituting for the 400-cc. beaker a 1-liter graduated flask, carrying a one-hole stopper fitted with a short glass tube drawn out to a point. Cool and add dropwise and with vigorous stirring 5 cc. less acid than before. Call the number of cubic centimeters used A. Grind up any small lumps with a glass rod flattened at one end, dilute to the mark with freshly boiled distilled water, close the flask with a solid stopper, mix thoroughly for 4 or 5 minutes and let settle for 30 minutes.

Pipette a 200-cc. portion, add phenolphthalein and titrate slowly with 0.5 N hydrochloric acid until the solution remains colorless on standing 1 minute. Call this additional number of cubic centimeters B. Then the percentage of available $\mathrm{CaO} = 2A + 5B$.

NOTES.

To secure accurate and concordant results, it is essential that this method be followed in minute detail.

For very accurate work 0.1 N acid may be used in place of the 0.5 N acid prescribed. The calculation then becomes: Available $\mathrm{CaO} = 2A + B$.

Standard acid solutions must be standardized by the U. S. Bureau of Standards benzoic acid No. 48-A through a sodium hydroxide solution.

PREPARATION OF STANDARD SOLUTION OF POTASSIUM PERMANGANATE.

Potassium Permanganate.

Prepare a solution of potassium permanganate containing 5.64 g. of the pure crystallized salt in each liter. Such a solution will be of such strength that 1 cc. will approximately equal 0.005 g. of CaO (Note 25).

The best and simplest way to prepare such a solution is to weigh out the required quantity of potassium permanganate and place in a bottle containing the proper volume of water a week or ten days before the solution is to be standardized. The bottle and its contents must be kept in a dark place and shaken occasionally for the first three or four days. When ready for standardization, siphon off the solution through a glass siphon into another bottle, leaving a depth of about one inch of solution undisturbed in the original bottle (Note 26). Thoroughly mix the solution in the second bottle by shaking and standardize as follows (Note 27):

Dissolve 0.5 g. of $Na_2C_2O_4$ (Note 28) in a 400-cc. beaker with 200 to 250 cc. of hot water (80 to 90° C.) and add 10 cc. of dilute H_2SO_4 (1 : 1) to the solution. Titrate at once with the permanganate solution being standardized, stirring vigorously and continuously. The permanganate solution must not be added more rapidly than 10 to 15 cc. per minute, and the last 0.5 to 1 cc. must be added dropwise, with particular care to allow each drop to be fully decolorized before the next is introduced. The excess of permanganate solution necessary to give the end point color is determined by running a blank determination in another beaker containing the same volume of acid and water present in the original beaker, and heated to the same temperature (Note 29). The value of the permanganate solution in grams of CaO per cubic centimeter of solution may be calculated from the following equations:

$$5Na_2C_2O_4 + 2KMnO_4 + 8H_2SO_4 = 5Na_2SO_4 + K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O.$$

$$5CaC_2O_4 + 2KMnO_4 + 8H_2SO_4 = 5CaSO_4 + K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O.$$

NOTES.

1. Ignitions in electric muffles are far superior to flame ignitions. If an electric muffle is not available, flame ignition and the blast lamp may be used.

2. If a platinum dish is not available, porcelain may be used. A glass container positively must *not* be used.

3. Instead of strong acid, that of half strength may be used and the heating begun at once.

4. For ordinary control work in the plant laboratory this correction may, perhaps, be neglected; the double evaporation must never be neglected.

5. Fusion with pyrosulfate is to be avoided on account of the subsequent introduction of sulfates into the solution.

6. This precipitate may also contain TiO_2, P_2O_5, and Mn_3O_4.

7. Where the iron is present in small quantities it is rather unsatisfactory to determine it in the ignited oxides from the 0.5-g. sample. Under these conditions the alternative method may be used.

8. The pyrosulfates are less troublesome and more effective than the bisulfates.

9. This correction for impurities should not be made when the hydrofluoric acid correction of the main silica determination has been omitted. After two evaporations, 1 to 2 mg. of silica are still to be found with the R_2O_3 precipitate.

10. Zinc will also reduce titanium. If this metal is to be determined and deducted from Al_2O_3, then H_2S must be used for reduction (boiling out the H_2S in a stream of CO_2 before titration). If titanium is not to be determined, the slight error introduced by its reduction with zinc is so small as to be negligible except in the most exact and detailed analyses.

11. The gravimetric method must be employed when a recovery of aluminum is desired, or when a determination of strontium is contemplated.

12. Care must be exercised in this washing, as 1000 cc. of boiling water will dissolve over 1 cg. of CaC_2O_4.

13. The volumetric method may be used for ordinary control work in the plant laboratory. It must be borne in mind that in this method all strontium oxide is reported as calcium oxide.

14. Use not more than 125 cc. for this washing.

15. The less the amount of magnesium present, the longer the precipitate must be allowed to settle.

16. Made by diluting NH_4OH with distilled water until the solution contains 2½ per cent NH_3 by weight. Three or four drops of HNO_3 (sp. gr. 1.42) are then added.

17. When manganese is present in the limestone, it will be caught in large part with the magnesium precipitate. (See U. S. Geological Survey *Bulletin 700*, p. 134.) In case manganese was not eliminated before this precipitation, the weighed pyrophosphate must be dissolved in HNO_3, tested for manganese with bismuthate, the manganese determined and deducted as $Mn_2P_2O_7$.

18. This determination may be made if desired upon the 0.5-g. sample ignited for solution in HCl. See first page of these methods under " Treatment of Sample."

19. With burned lime and hydrated lime this determination may be omitted. Even when made in an atmosphere free from CO_2 the dried sample absorbs CO_2 sufficiently rapidly during weighing to make the result of questionable value.

20. Boiling for one minute is usually sufficient.

21. In limestones very low in phosphorus it may be deemed desirable to concentrate the phosphorus from a considerable weight of sample. In this case, proceed as directed by F. Hinden (*Zeitschr. Anal. Chemie*, Vol. 54, p. 214 (1915)), as follows: To the nitric acid solution of the large sample add a few drops of Fe_2Cl_6 solution and then NH_4OH until the liquid becomes slightly turbid. Then add 0.5 g. of pure precipitated $CaCO_3$ and boil for five minutes. Filter, dissolve the precipitate in dilute HNO_3 and proceed as above.

22. Titanium and vanadium, which interfere in this determination, should not be encountered in appreciable amounts in this class of material.

Instead of the specified alkalimetric titration method, the phosphorus in the phosphomolybdate may be determined as magnesium pyrophosphate (U. S. Geological Survey *Bulletin 700*, pp. 178–179), or by the reductor method (A. A. Blair, "Chemical Analysis of Iron," 8th Edition, pp. 91–92).

23. The persulfate method may be employed if desired.

24. The separation of manganese with the R_2O_3 precipitate by the use of bromine is not recommended on account of the incompleteness of the precipitation. Separation of manganese by means of ammonium sulfide after the ammonia precipitation is also undesirable on account of the introduction of the reagent and the slimy character of the precipitate.

See also Note 17 concerning the contamination of magnesium pyrophosphate by manganese pyrophosphate and the need of corrective measures when manganese is present in the material under analysis.

25. With high calcium limes, more than 50 cc. of this solution will probably be required to titrate the liberated oxalic acid. In such cases the use of a larger burette is to be preferred to increasing the strength of the permanganate solution.

26. The siphon should not extend nearer than 1 in. from the bottom of the original bottle.

27. This solution should be standardized at least once every week. It will be found more convenient to make a solution as above described and standardize it every week, than to attempt, by boiling and filtering, to make a solution which will not change.

28. Sodium oxalate for standardization of potassium permanganate solution should be purchased from the U. S. Bureau of Standards, Washington, D. C. This material is sold by the Bureau of Standards as Sample No. 40a.

29. When the end point is reached, the temperature of the solution should not be below 60° C.

TENTATIVE METHOD
OF
SECURING SPECIMENS OF HARDENED CONCRETE FROM THE STRUCTURE[1]

Serial Designation: C 42 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921; Revised, 1925.

Scope. 1. The methods and precautions herein specified apply to securing test specimens from hardened concrete in the structure.

Precautions. 2. A specimen from hardened concrete to be tested for strength shall not be taken until the concrete has become hard enough so that the cutting of the specimen will not disturb the bond between the mortar content and the coarse aggregate of the specimen.

Apparatus. 3. The specimen from the structure can best be secured by use of a core drill. For specimens taken perpendicular to a horizontal surface, a drill using chilled shot may be used; but when taken perpendicular to a vertical surface, a diamond drill should be used.

Test Specimen. 4. (*a*) The core specimen taken shall be as nearly as possible a cylinder whose length is twice the diameter.

(*b*) In securing a specimen perpendicular to a horizontal surface care shall be taken to secure, if possible, a specimen whose beds shall be parallel to the horizontal bed of the concrete as originally placed.

(*c*) In securing a specimen perpendicular to a vertical surface, or to a surface with a batter, care shall be taken as to the point of securing the specimen. The lower portion of any one unit of depositing in mass concrete is more dense than is the upper portion of the unit, therefore a specimen shall be taken from near the middle of such unit of deposit.

Procedure. 4. A specimen to be taken from hardened concrete which has been removed from a structure shall be cut out by a drill or shall be cut into a symmetrical test-piece by tooling or by sawing. The method of sawing can seldom be used to advantage in the field. In selecting this test specimen care shall be used to see that the concrete selected has not been injured or shattered by the method of its removal from the structure.

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. J. C. Pearson, Secretary of Committee C-9 on Concrete and Concrete Aggregates, Lehigh Portland Cement Co., Allentown, Pa.

Ends. 5. The specimens secured shall have ends, or beds, as nearly as possible perpendicular to its axis, and, if the specimen has ends with uneven surfaces, these ends shall be made to parallel plane surfaces with a mortar richer than the mortar of the specimen or with a mixture of cement and calcined gypsum (plaster of Paris).

6. In case the dimensions of a specimen are necessarily appreciably different from the recommended shape which requires a height twice the diameter, allowance for the ratio of height to diameter must be made in determining the strength. When the height is more than twice the diameter, that is, when the ratio is greater than 2, no correction need be made. For other ratios corrections should be made as follows:

Ratio of Height of Cylinder To Diameter, $\frac{h}{d}$					
1.75	multiply	crushing	strength	by	0.98
1.50	"	"	"	"	0.95
1.25	"	"	"	"	0.94
1.10	"	"	"	"	0.90
1.00	"	"	"	"	0.85
0.75	"	"	"	"	0.70
0.50	"	"	"	"	0.50

The direction of the application of the load in the compression test shall be stated with reference to the direction of the compaction of the mass of concrete in the structure, and where it is practicable the compression load shall be applied in the same direction. In order that the tests be made under uniform conditions as to moisture content, the test specimens shall be completely submerged in water for 48 hours and the compression test shall be made immediately thereafter.

TENTATIVE DEFINITIONS OF TERMS RELATING TO THE GYPSUM INDUSTRY[1]

Serial Designation: C 11 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to **annual** revision.

ISSUED, 1916; REVISED, 1921, 1922, 1923, 1924, 1925.

Accelerator.—Any material or substance which, when used in stucco, plaster, mortar, concrete, etc., will hasten set beyond the limit of testing error.

Aggregate.—Any inert material used as a filler in stucco, plaster, mortar, concrete, etc., without regard to its function as a binding material.

Binder.—Any fibrous material which will increase the cohesiveness of plaster and stucco while in its plastic state.

Cement.—Any material, or a mixture of materials (without aggregate) which becomes plastic upon the addition of water and, when in a plastic state, possesses adhesive and cohesive properties, and hardens in place. The word "cement" is used without regard to the composition of the material and does not define its use or location of use as do the words "stucco," "plaster" and "mortar."

Consistency.—The degree of wetness of a plastic mixture.

Calcined Gypsum.—Gypsum partially dehydrated by means of heat, having the chemical formula $CaSO_4 \cdot \frac{1}{2}H_2O$.

Crushed Gypsum.—Gypsum subjected to a primary crushing operation.

Gypsum.—Calcium sulfate, combined with two molecules of water, in crystallin form, having the chemical formula $CaSO_4 \cdot 2H_2O$.

Gypsum Plastering Sand.—The fine granular material naturally or artificially produced by the disintegration of rock containing not less than 80 per cent by weight of silica, feldspar, dolomite, magnesite or calcite.

Gypsum Plaster Board.—A sheet composed of an incombustible core of gypsum, surfaced with paper or other fibrous material which is firmly bonded to the core, or with intermediate layers of such material within the core, or an incombustible sheet of gypsum with not more than 15 per cent by weight of fiber intimately mixed; and designed to be used as a lath for the reception of gypsum plaster.

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[1] Criticisms of these Tentative Definitions are solicited and should be directed to Mr. W. E. Emley, Chairman of Committee C-11 on Gypsum, Chemist, U. S. Bureau of Standards.

Gypsum Wall Board.—A sheet composed of an incombustible core of gypsum, surfaced with paper or other fibrous material which is firmly bonded to the core, and designed to be used, without the addition of plaster, for walls, ceilings or partitions and affording a surface suitable to receive decoration.

Lath.—A material whose primary function is that of a base or background for the reception of plaster or stucco. The word "lath" is used without regard to the composition of the material, defining only its use.

Mortar.—Any material used in a plastic state that can be trowelled, and becomes hard in place, uniting masonry materials. The word "mortar" is used without regard to the composition of the material, defining its use as a bonding material, as contrasted with the words "stucco" and "plaster."

Plaster.—Any material used in a plastic state to form a hard covering for the interior surfaces, walls and ceilings, etc., of any building or structure. The word "plaster" is used without regard to the composition of the material, defining only its use and location of use, as contrasted with the words "stucco" and "mortar."

Plasticity.—The yielding property of a wet mixture to change of form.

Retarder.—Any material or substance which, when used in stucco, plaster, mortar, concrete, etc., will retard set beyond the limit of testing error.

Sized Gypsum.—Crushed gypsum of prescribed diameter of individual particles.

Stucco.—Any material used in a plastic state to form a hard covering for the exterior walls or other exterior surfaces of any building or structure. The word "stucco" is used without regard to the composition of the material, defining only its use and location of use, as contrasted with the words "plaster" and "mortar."

Wood Fiber.—A material produced by grinding or shredding wood.

TENTATIVE DEFINITION OF THE TERM SAND[1]

Serial Designation: C 58-25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924; REVISED, 1925.

Sand.—The fine granular material resulting from the natural disintegration or the crushing of rock or slag.

NOTE.—When used without a qualifying adjective it is generally understood to mean a product of the natural disintegration of siliceous or calcareous rock. The size of particle and other physical characteristics should be taken care of in specifications and therefore no limits are mentioned in the definition.

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[1] Criticisms of this Tentative Definition are solicited and should be directed to Mr. L. R. Ferguson, Chairman of the Sub-Committee, of Committee E-8, on Sand, International Cement Corporation, 342 Madison Ave., New York City.

TENTATIVE SPECIFICATIONS
FOR
RAW TUNG OIL[1]

Serial Designation: D 12 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1923, 1925.

I. PROPERTIES AND TESTS.

Properties. 1. Raw tung oil shall conform to the following requirements:

	MAXIMUM.	MINIMUM.
Specific gravity at 15.5°/15.5° C	0.943	0.9400
Acid number (Alcohol-Benzol)	8.0	.....
Saponification number	195	190
Unsaponifiable matter, per cent	0.75	.....
Refractive index at 25° C	1.520	1.5165
Iodine number (Wijs)	.....	163
Heating test, minutes	12	....

II. METHODS OF TESTING.

SOLUTIONS REQUIRED.

Solutions Required. 2. The following reagents will be required:

Standard Sodium Thiosulfate Solution.—Dissolve pure sodium thiosulfate in distilled water that has been well boiled to free it from carbon dioxide in the proportion so that 24.83 g. crystallized sodium thiosulfate will be present in 1000 cc. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Treadwell-Hall, *Analytical Chemistry*, Vol. II, third edition, p. 646.) This solution will be approximately N/10 and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly N/10 strength. Preserve in a stock bottle with a guard tube filled with soda lime.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

These Tentative Specifications are in effect a revision of the Standard Specifications for Purity of Raw Tung Oil. The Standard Specifications, which were last published under the Serial Designation: D 12 – 16, have accordingly been withdrawn.

Starch Solution.—Stir up 2 to 3 g. of potato starch or 5 g. soluble starch with 100 cc. of 1-per-cent salicylic acid solution, add 300 to 400 cc. boiling water, and boil the mixture until the starch is practically dissolved. Dilute to 1 liter.

Potassium Iodide Solution.—Dissolve 150 g. of potassium iodide free from iodate in distilled water and dilute to 1000 cc.

Iodine-Monochloride Solution.—Dissolve iodine in glacial acetic acid that has a melting point of 14.7 to 15° C. and is free from reducing impurities in the proportion so that 13 g. of iodine will be present in 1000 cc. of the solution. The preparation of the iodine-monochloride solution presents no great difficulty, but it shall be done with care and accuracy in order to obtain satisfactory results. There shall be in the solution no sensible excess either of iodine or more particularly of chlorine, over that required to form the monochloride. This condition is most satisfactorily attained by dissolving in the whole of the acetic acid to be used the requisite quantity of iodine, using a gentle heat to assist the solution, if it is found necessary. Set aside a small portion of this solution, while pure, and pass dry chlorine into the remainder until the halogen content of the whole solution is doubled. Ordinarily it will be found that by passing the chlorine into the main part of the solution until the characteristic color of free iodine has just been discharged, there will be a slight excess of chlorine, which is corrected by the addition of the requisite amount of the unchlorinated portion until all free chlorine has been destroyed. A slight excess of iodine does little or no harm, but excess of chlorine must be avoided.

Chloroform.—Should be U. S. P.

Standard Sodium Hydroxide Solution.—Prepare a stock concentrated solution of sodium hydroxide by dissolving NaOH in water in the proportion of 200 g. NaOH to 200 cc. water. Allow this solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the precipitate of sodium carbonate into another clean bottle. Add clear barium hydroxide solution until no further precipitate forms. Again allow to settle until clear. Draw off about 175 cc. and dilute to 10 liters with freshly boiled distilled water. Preserve in a stock bottle provided with a large guard tube filled with soda lime. Determine the exact strength by titrating against pure benzoic acid (C_6H_5COOH) using phenolphthalein as indicator. (See Bureau of Standards *Scientific Paper No. 183.*) This solution will be approximately N/4, but do not attempt to adjust it to any exact value. Determine its exact strength and make proper corrections in using it.

Alcoholic Sodium Hydroxide Solution.—Dissolve pure NaOH in 95-per-cent ethyl alcohol in the proportion of about 22 g. per 1000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle, and keep well stoppered. This solution should be colorless or only slightly yellow when used; it will keep colorless longer if the alcohol is previously treated with NaOH (about 80 g. to 1000 cc.), kept at about 50° C. for 15 days, and then distilled. For an alternate method see *Journal*, American Chemical Society, 1906, p. 395.

Half Normal Sulfuric Acid Solution.—Add about 15 cc. H_2SO_4 (sp. gr. 1.84) to distilled water, cool and dilute to 1000 cc. Determine the exact strength by titrating against freshly standardized NaOH or by any other accurate method. Either adjust to exactly N/2 strength or leave as originally made, applying appropriate correction.

METHODS.

Methods of Testing.

3. The oil shall be tested in accordance with the following methods:

General.—The laboratory sample shall be thoroughly mixed by shaking, stirring, or pouring from one vessel to another and the samples for the individual tests taken from this thoroughly mixed sample.

Specific Gravity.—Use a pyknometer accurately standardized and having a capacity of at least 25 cc., or any other equally accurate method, making the test at 15.5° C., water being 1 at 15.5° C.

Acid Number.—Weigh from 5 to 10 g. of the oil. Transfer to a 300-cc. Erlenmeyer flask. Add 50 cc. of a mixture of equal parts by volume of 95-per-cent ethyl alcohol and c. p. reagent benzol. (*This mixture should be previously titrated to a very faint pink with dilute alkali solution, using phenolphthalein as an indicator*). Add phenolphthalein indicator and titrate at once to a faint permanent pink color with the standard sodium hydroxide solution. Calculate the acid number (milligrams KOH per gram of oil).

Saponification Number.—Weigh about 2 g. of the oil in a 300-cc. Erlenmeyer flask. Add 25 cc. alcoholic sodium hydroxide solution. Put a condenser loop inside the neck of the flask and heat on the steam bath for one hour. Cool, add phenolphthalein as indicator, and titrate with N/2 H_2SO_4. Run two blanks with the alcoholic sodium hydroxide solution. These should check within 0.1 cc. N/2 H_2SO_4. From the difference between the number of cubic centimeters of N/2 H_2SO_4 required for the blank and for the determination, calculate the saponification number (milligrams KOH required for 1 g. of the oil).

Unsaponifiable Matter.—Weigh 8 to 10 g. of the oil. Transfer to a 250-cc. long-neck flask. Add 5 cc. of strong solution of sodium hydroxide (equal weights of NaOH and H_2O), and 50 cc. 95-per-cent ethyl alcohol. Put a condenser loop inside the neck of the flask and boil for two hours. Occasionally agitate the flask to break up the liquid but do not project the liquid onto the sides of the flask. At the end of two hours remove the condenser and allow the liquid to boil down to about 25 cc.

Transfer to a 500-cc. glass-stoppered separatory funnel, rinsing with water. Dilute with water to 250 cc., add 100 cc. redistilled ether. Stopper and shake for one minute. Let stand until the two layers separate sharp and clear. Draw all but one or two drops of the aqueous layer into a second 500-cc. separatory funnel and repeat the process, using 60 cc. of ether. After thorough separation draw off the aqueous solution into a 400-cc. beaker, then the ether solution into the first separatory funnel, rinsing down with a little water. Return the aqueous solution to the seond separatory funnel and shake out again with 60 cc. of ether in a similar manner, finally drawing the aqueous solution into the beaker and rinsing the ether into the first separatory funnel.

Shake the combined ether solution with the accumulated water rinsings and let the layers separate sharp and clear. Draw off the water and add it to the main aqueous solution. Shake the ether solution with two portions of water (about 25 cc. each). Add these to the main water solution.

Swirl the separatory funnel so as to bring the last drops of water down to the stopcock, and draw off until the ether solution just fills the bore of the stopcock. Wipe out the stem of the separatory funnel with a bit of cotton on a wire. Draw the ether solution (portionwise if necessary) into a 250-cc. flask and distill off. While still hot, drain the flask into a small weighed beaker, rinsing with a little ether. Evaporate this ether, cool and weigh.

NOTE.—The unsaponifiable oil from adulterated drying oils is volatile and will evaporate on long heating. Therefore heat the beaker on a warm plate, occasionally blowing out with a current of dry air. Discontinue heating as soon as the odor of ether is gone.

Refractive Index.—Use a properly standardized Abbé refractometer at 25° C., or any other equally accurate instrument.

Iodine Number.—Place a small quantity of the sample in a small weighing burette or beaker. Weigh accurately. Transfer by dropping from 0.16 to 0.19 g. to a 500-cc. bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine tests. Reweigh the burette or beaker and determine the amount of sample used. Add 10 cc. of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc. of chloroform to each of two empty bottles like that used for the sample. Add to each bottle 25 cc. of the

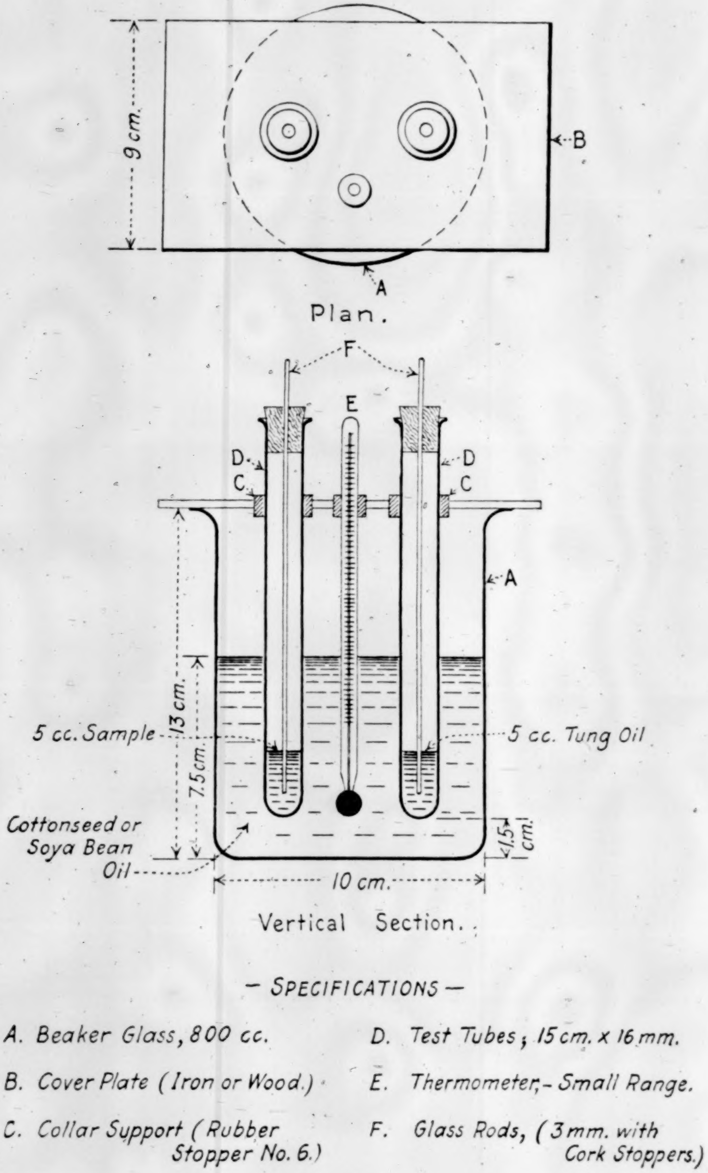
9 cm.
B
A
Plan.
F
E
D
C
D
C
A
13 cm.
5 cc. Sample
7.5 cm.
5 cc. Tung Oil
Cottonseed or Soya Bean Oil
1.5 cm.
10 cm.
Vertical Section.
– SPECIFICATIONS –
A. Beaker Glass, 800 cc.
B. Cover Plate (Iron or Wood.)
C. Collar Support (Rubber Stopper No. 6.)
D. Test Tubes; 15 cm. x 16 mm.
E. Thermometer, - Small Range.
F. Glass Rods, (3 mm. with Cork Stoppers.)

FIG. 1.—Tung Oil Heat Test Apparatus (Revised 1920).

NOTE: Collars *C* may be omitted and tubes supported in present place by aid of wire gauze placed in bottom of oil bath and resting on bottom of beaker.

Wijs solution and let stand with occasional shaking for 30 minutes in a dark place at a temperature of from 21 to 23° C. Add 10 cc. of the 15-per-cent potassium iodide solution and 100 cc. of water, and titrate with standard sodium thiosulfate using starch as the indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titration and the titration on the samples and the iodine value of the thiosulfate solution, calculate the iodine number of the samples tested. (Iodine number is given in centigrams of iodine to 1 g. of sample.)[1]

Heating Test.—Test tubes containing the oil should be 15 cm. by 16 mm., with a mark near the bottom to indicate 5 cc., and closed by a cork so perforated that a glass rod 3 mm. in diameter can move freely.

Fill an 800-cc. glass beaker (height, 13 cm.; diameter, 10 cm.). with cottonseed oil or soya bean oil to a height of 7.5 cm. Place a thermometer so as to be 1.5 cm. from the bottom of the bath. (See Fig. 1.)

Use a nitrogen-filled, chemical thermometer; engraved stem; total length 4 to 4½ in., graduated from 210 to 310° C. in 2° intervals; the length between 210 and 310° C. not less than 2½ in. Thermometer glass shall be well annealed.

When the bath temperature is 293° C. (560° F.) and very slowly rising at this point, place the tube containing 5 cc. of the oil to be tested and the tube containing 5 cc. of a control sample of known value, so that the bottom of each tube is level with the lowest part of the bulb of the thermometer. If desired, the collars *C* may be omitted and the tubes allowed to rest upon a piece of wire gauze placed in the bottom of the oil bath so that the tubes will be 1.5 cm. from the bottom of the bath. Note the time. Remove the source of heat for about 45 seconds and then reapply. Before 2 minutes have elapsed the temperature of the bath will have fallen to 282° C. (530° F.), at which point it should be kept as steady as possible. When the samples have been in the bath 9 minutes, raise the glass rods at intervals of ¼ minute. Note the time when each sample becomes firmly set. At this period the oil will be so stiff that the entire tube may be lifted by aid of the rod if the collar *C* is omitted from the apparatus. As setting or jellying takes place within a few seconds of fluidity, a good end determination is afforded. Remove the specimens. Heat the bath again to 293° C., and repeat the experiment with fresh portions of the sample.

No stirrer is used in the bath. A screen around the bath enables the temperature to be more easily reached. When the bath oil has become tarry and viscid, it should be renewed; otherwise heating may be irregular.

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[1] It is always well to include a test on a sample of tung oil of known iodine value. This may be kept in a dark-colored bottle as a standard.

Quality Test.[1]—Into an ordinary agateware casserole, having a bottom diameter of 3 in., weigh 150 g. of the tung oil to be tested, and set the casserole on a wide-flanged tripod having a 3-in. opening. The object of the flange is to prevent super-heating of the sides of the casserole. Heat rapidly with a full Bunsen flame, stirring with a thermometer, until the heat reaches 540° F. (282.2° C.). Turn down the flame and hold the heat as near 540° F. (282.2° C.) as possible, stirring with the thermometer, until on lifting the latter the oil drops with a pronounced string, showing that polymerization has started. The time required after reaching 540° F. (282.2° C.) until the string is noted, is the time of the heat test. For pure tung oils this will not exceed eight minutes. As soon as the oil strings, remove the lamp and the thermometer, and stir with a stiff spatula until the oil is solid. After stringing, a pure tung oil will require not over 40 seconds more to become solid. When solid, allow to stand just one minute, then turn out, upside down, on clean paper and cut with a clean spatula. Pure tung oil gives a gel that is dry, not adhering to the spatula when cut, that is firm, crumbling under pressure of the spatula without sticking, and the cut portions should crumble under pressure like dry bread crumbs. Adulterated tung oil gives a gel that is soft, sticky, and which will not crumble.

III. SAMPLING

The method of sampling given under Method (*a*) below shall be used whenever it is feasible to apply it. To meet conditions when (*a*) is not applicable, method (*b*) or (*c*) shall be used, according to the special conditions that obtain.

(*a*) *During Loading of Tank Cars or Filling of Containers for Shipment.*—

The purchaser's inspector shall draw a sample at the discharge pipe where it enters the receiving vessel or vessels. The total sample shall be not less than 1 gal. per each tank car or 10,000 gal. equivalent, and shall be a composite of small samples of not more than 1 pt. each taken at regular intervals during the entire period of loading or filling.

The sample thus obtained shall be thoroughly mixed, and from this composite sample three portions of not less than 1 qt. each shall be placed in clean, dry glass bottles or tin cans, which must be filled with the sample and securely stoppered with new clean corks or well-fitting metal covers or caps. These shall be sealed and labeled distinctly by the inspector, one delivered to the buyer, one to the seller, and the other held for check in case of dispute.

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[1] Furnished by R. S. Worstall.

(*b*) *From Loaded Tank Cars or Other Large Vessels.*—

The total sample shall be not less than 1 gal. per each tank car or 10,000 gal. equivalent, and shall be a composite of numerous small samples of not more than 1 pt. each, taken from the top, bottom, and intermediate points by means of a glass or metal container with removable stopper or top. This device attached to a suitable pole is lowered to various desired depths when the stopper or top is removed and the container allowed to fill. The sample thus obtained shall be handled as in Method (*a*). For large shipments in freighters ranging from 400 to 1000 tons, first draw samples from the top and bottom of both port and starboard sections of the tank. These samples shall be visually examined, and if the general appearance gives indications that the oil is satisfactory, pumping shall be started and 1-pt. samples drawn from a bleeder in the discharge line at least once in every thirty minutes, so that the total mixed sample amounts to as many gallons as there are units of 10,000 gal. in the cargo. These individual samples are composited to make one uniform sample representative of the entire cargo and this is handled as in Method (*a*).

(*c*) *Barrels and Drums.*—

Not less than 10 per cent of the packages in any shipment or delivery of barrels and drums shall be sampled. The packages shall be shaken, rolled, and stirred to thoroughly mix the contents. The samples from the individual containers shall be taken through the bung hole or holes not less than 1¼ in. in diameter bored in the head or side for the purpose. The apparatus for drawing the sample shall consist of a glass tube (known as a thief) about 1 in. in diameter and somewhat longer than the length or diameter of the oil container, a conical stopper that will fit the glass tube and is not more than ½ in. in length, fastened to a stiff metal rod not more than ¼ in. in diameter and not less than 4 in. longer than the glass tube. The stopper is lowered by the rod until it rests on the bottom of the cask, the tube slipped down slowly over the rod, and finally pressed on the stopper. By holding the tube and the rod, the column of oil can then be removed. This process is repeated until the required amount of sample is obtained, which shall be not less than 1 gal. This is mixed and handled as in Method (*a*). During the winter when the oil is very often in a solid condition, the same procedure should be followed, replacing the thief with a tallow trier, which is essentially a graduated piece of steel tubing semi-circular in cross-section and about 3 ft. long. This is pushed into the oil, turned a few times and withdrawn, thereby removing a solid core of oil.

TENTATIVE SPECIFICATIONS
FOR
DRY BLEACHED SHELLAC[1]

Serial Designation: D 207 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925

I. GRADES

Grades. 1. Dry bleached shellac[2] shall be furnished in two grades, namely, regular and refined.[3]

II. PROPERTIES AND TESTS

Properties. 2. Dry bleached shellac shall conform to the following requirements:

	Regular	Refined
Iodine number, maximum	10	10
Matter insoluble in hot alcohol, maximum	1.0 per cent	0.2 per cent
Moisture, maximum	5.0 "	5.0 "
Wax, maximum	5.5 "	0.2 "
Matter soluble in water, maximum	1.0 "	0.3 "
Ash, maximum	1.0 "	0.3 "

III. METHODS OF TESTING

3. The determination of iodine number, matter insoluble in hot alcohol and moisture shall be made in accordance with the Standard Methods of Testing Shellac (Serial Designation: D 29) of the American Society for Testing Materials.[4]

4. The determination of wax shall be made in accordance with the Standard Method of Test for Determination of Wax in Shellac

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

[2] This material is white shellac. It is also known as "bonedry," "vacdry" or "kilndried" bleached shellac.

[3] Refined bleached shellac has been treated in the process of manufacture so as to remove practically all wax and matter insoluble in hot alcohol.

[4] 1924 Book of A.S.T.M. Standards.

(Serial Designation: D 29) of the American Society for Testing Materials.[1]

5. The determination of matter soluble in water shall be made as follows:

Ten to twenty-five grams of dry shellac shall be ground so that 100 per cent shall pass through a No. 20 sieve (840 micron opening). The shellac shall be placed in a 250-cc. beaker or flask and 100 cc. of distilled water added. It shall be stirred thoroughly, covered with a watch glass and permitted to stand at room temperature (21° C.) for four hours, with occasional stirring.

It shall be decanted through a 12.5-cm. filter paper into a weighed evaporating dish, washing the shellac and filter with at least 50 cc. more of water. It shall be evaporated and the extract dried at 105° C. for one hour or more to constant weight. The increase in weight shall be reported as the water-soluble extract.

6. The determination of ash shall be made as follows:

Approximately 2 to 3 g. of the sample shall be accurately weighed, transferred to a weighed porcelain or platinum crucible and ignited at as low a temperature as possible until all organic matter has been destroyed. The crucible and contents shall be cooled and weighed and the percentage of ash calculated.

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[1] A.S.T.M. Standards Adopted in 1925.

TENTATIVE SPECIFICATIONS
FOR
IRON OXIDE AND IRON HYDROXIDE[1]

Serial Designation: D 84 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1924, 1925.

Scope. 1. These specifications cover iron oxide and iron hydroxide pigments of red and brown colors.

I. MANUFACTURE.

Process. 2. (*a*) *Dry Pigment.*—The pigment shall be very finely ground iron oxide or iron hydroxide or a mixture thereof. Siliceous minerals may be present and, if necessary, sufficient carbon pigment may be added to produce the desired color.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

II. PROPERTIES AND TESTS.

Composition and Properties. 3. (*a*) *Dry Pigment.*—The pigment shall conform to the following requirements:

	MAXIMUM.	MINIMUM.
Ferric oxide, per cent	...	30.0
Material other than ferric oxide, insoluble siliceous matter and loss on ignition, per cent	10.0	...
Coarse particles retained on a Standard No. 325 screen,[2] per cent	3.0	...

Organic coloring matters shall be absent.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

[2] For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.) the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

The color and color strength, when specified, and the drying time with a vehicle which has been mutually agreed on, shall be equal to those of an agreed sample.

(*b*) *Paste.*—The paste as received and three months thereafter shall not be caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily with linseed oil, turpentine or volatile mineral spirits, or any combination of these, without curdling.

The paste shall conform to the following requirements:

	MAXIMUM.	MINIMUM.
Pigment, per cent	72.0	68.0
Linseed oil, per cent	32.0	28.0
Moisture and other volatile matter, per cent	0.7	....
Coarse particles and skins (total residue retained on a Standard No. 325 screen,[1] based on pigment), per cent	3.5	....

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

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[1] For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS
FOR
LITHOPONE[1]

Serial Designation: D 208 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925.

Scope.

1. These specifications cover the pigment consisting of zinc sulfide and barium sulfate, commercially known as lithopone. The pigment may be purchased in the dry form or ground in oil to form a paste.

I. MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be made by suitable treatment of a mixture of precipitated zinc sulfide and precipitated barium sulfate.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

II. PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The behavior on exposure to light, the mixing properties with an approved vehicle, the final consistency with this vehicle and the color shall be equal to, and the brightness and tinting strength (or hiding power) shall be not less than, those of a sample, mutually agreed on by buyer and seller.

(*b*) *Dry Pigment.*—The dry pigment shall meet the following requirements:

	Maximum	Minimum
Coarse particles and skins (total residue retained on a standard No. 325 screen), per cent........	1.0	....
Zinc sulfide, per cent...........................	...	26.0
Zinc oxide, per cent............................	1.0	....
Material soluble in water, per cent.............	0.8	....
Barium sulfate..................................	The remainder	

(*c*) *Paste.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances. The paste shall meet the following requirements:

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

	MAXIMUM	MINIMUM
Pigment, per cent	80.0	76.0
Linseed oil, per cent	24.0	20.0
Coarse particles and skins (total residue retained on a standard No. 325 screen, based on pigment), per cent	1.5	....

Number of Tests.

4. (*a*) *Paste.*—One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

(*b*) *Dry Pigment.*—Dry pigment shall be sampled by taking a portion from every tenth barrel or from every one hundredth bag of the dry pigment.

NOTE.—For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS
FOR
LAMPBLACK[1]

Serial Designation: D 209 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

I. MANUFACTURE

Scope.

1. These specifications cover the pigment commonly known as lampblack. The pigment may be purchased in the dry form, or ground in oil or in japan to form a paste.

Process.

2. (*a*) The dry pigment shall be made by burning oils or tars in such a manner as to form a deposit of carbon or soot. The pigment shall be high grade in every respect, shall be free from oil, greasy matter and from admixture of any other substance.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan.

II. PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and tone shall be equal to, and the tinting strength not less than that of a sample mutually agreed on by buyer and seller.

(*b*) *Dry Pigment.*—The dry pigment shall meet the following requirements:

Coarse particles retained on a standard No. 325 screen, maximum, per cent	1.0
Ash, maximum, per cent	1.0
Benzol extract (which must be colorless) maximum, per cent	0.5
Tone when diluted with zinc oxide	Clear-blue-gray

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances. The paste shall meet the following requirements:

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

	MAXIMUM	MINIMUM
Pigment, per cent	40	25
Linseed oil, per cent	75	60
Material volatile at 105° C	0.7	..
Coarse particles and skins left on a No. 325 screen (per cent of the dry pigment)	1.0	..

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard, flat coat. It shall meet the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	40	25
Vehicle, per cent	75	60
Coarse particles and skins retained on a standard No. 325 screen (per cent of the dry pigment)	1.0	..
Non-volatile matter in the vehicle, per cent of the vehicle	..	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

NOTE.—For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.) the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS
FOR
BONE BLACK[1]

Serial Designation: D 210 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

Scope.

1. These specifications cover the pigment commonly known as bone black, ivory black or drop black. It may be purchased in the dry form, or ground in oil or in japan to form a paste.

I. MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The dry pigment shall be made by the calcination of bones and shall be unmixed with any other substance.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment in pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the pigment in high-grade grinding japan.

II. PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and tone shall be equal to, and the tinting strength not less than, that of a sample mutually agreed on by buyer and seller.

(*b*) *Dry Pigment.*—The dry pigment shall meet the following requirements:

Coarse particles retained on a standard No. 325 screen, maximum, per cent	2.0
Ash, maximum, per cent	85.0
Ash insoluble in acids, maximum, per cent	1.0
Benzol extract (which must be colorless), maximum, per cent	0.5
Tone when diluted with zinc oxide	Clear-blue-gray

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances. The paste shall meet the following requirements:

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

	MAXIMUM	MINIMUM
Pigment, per cent	55	45
Linseed oil, per cent	55	45
Coarse particles and skins (total residue retained on a standard No. 325 screen, based on pigment), per cent	2.5	..

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard, flat coat. It shall meet the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	55	45
Vehicle (japan), per cent	55	45
Coarse particles and skins (total residue retained on a standard No. 325 screen, based on pigment), per cent	2.5	..
Non-volatile matter in the vehicle, per cent of the vehicle	..	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

NOTE.—For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS
FOR
CHROME YELLOW[1]

Serial Designation: D 211 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting **criticism** and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

Scope. 1. These specifications cover the pigments commonly known as lemon chrome yellow, medium chrome yellow, and orange chrome yellow. The pigment may be purchased in the dry form or ground in oil, or in japan, to form a paste.

I. MANUFACTURE

Process. 2. (*a*) *Dry Pigments.*—The dry pigments, lemon chrome yellow and orange chrome yellow, shall be chemical precipitates consisting of normal or basic lead chromates or mixtures of these with or without admixtures of other insoluble compounds of lead, but without any other admixtures.

Dry medium chrome yellow shall be chemically precipitated normal lead chromate.

(*b*) *Pastes in Oil.*—The pastes in oil shall be made by thoroughly grinding the specified pigments with pure raw or refined linseed oil.

(*c*) *Pastes in Japan.*—The pastes in japan shall be made by thoroughly grinding the specified pigments in high-grade grinding japan.

II. PROPERTIES AND TESTS

Composition and Properties. 3. (*a*) The color and tone shall be equal to, and the tinting strength not less than, that of a sample mutually agreed on by buyer and seller.

(*b*) The dry pigments shall meet the following requirements:

	LEMON CHROME ORANGE CHROME	MEDIUM CHROME
Total matter soluble in water, maximum, per cent	0.5	0.5
Total of all substances other than insoluble compounds of lead, maximum, per cent	3.0	3.0
Organic colors or lakes	None	None

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

	LEMON CHROME ORANGE CHROME	MEDIUM CHROME
Normal lead chromate ($PbCrO_4$), minimum, per cent	..	97.0
Coarse particles (total residue on a No. 325 screen), per cent, not over	1.0	1.0

(c) *Paste in Oil.*—The pastes as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. They shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances.

The pastes shall meet the following requirements:

	LEMON CHROME ORANGE CHROME		MEDIUM CHROME	
	MAXIMUM	MINIMUM	MAXIMUM	MINIMUM
Pigment, per cent	85	65	82	75
Linseed oil, per cent	35	15	25	18
Water and other volatile matter, per cent	0.7	..	0.7	..
Coarse particles and skins (total residue retained on a No. 325 screen), per cent of the pigment	1.5	..	1.5	..

(d) *Paste in Japan.*—The pastes as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard flat coat. They shall meet the following requirements:

	LEMON CHROME ORANGE CHROME		MEDIUM CHROME	
	MAXIMUM	MINIMUM	MAXIMUM	MINIMUM
Pigment, per cent	85	65	82	75
Vehicle (japan) per cent	35	15	25	18
Coarse particles and skins (total residue retained on a No. 325 screen), per cent of the pigment	1.5	..	1.5	..
Non-volatile matter in the vehicle, per cent of the vehicle	..	40	..	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

NOTE.—For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS
FOR
PURE CHROME GREEN[1]

Serial Designation: D 212 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

Scope.

1. These specifications cover what is commercially known as pure chrome green. The pigment may be purchased in the dry form or ground in oil or in japan to form a paste.

I. MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be a co-precipitated mixture of lead chromate and iron ferro and ferricyanide blue, with or without other insoluble compounds of lead.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment with high-grade grinding japan.

II. PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and tone shall be equal to, and the tinting strength not less than, that of a sample mutually agreed on by buyer and seller.

(*b*) *Dry Pigment.*—The dry pigment shall meet the following requirements:

Percentage of total lead present in the form of chromate	75
Total impurities, maximum, per cent	3
Coarse particles (total residue retained on a No. 325 screen), maximum, per cent	1.0

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances. The paste shall meet the following requirements:

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

	MAXIMUM	MINIMUM
Pigment, per cent	75	65
Linseed oil, per cent	35	25
Coarse particles and skins (total residue retained on a No. 325 screen), per cent of the pigment	1.5	..

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard, flat coat. The paste shall meet the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	75	65
Vehicle (japan), per cent	35	25
Coarse particles and skins (total residue retained on a No. 325 screen), per cent of the pigment	1.5	..
Non-volatile matter in vehicle, per cent of the vehicle	..	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

NOTE.—For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS
FOR
REDUCED CHROME GREEN[1]

Serial Designation: D 213 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

Scope.

1. These specifications cover what is known commercially as reduced chrome green, also known as grinders green. The pigment may be purchased in the dry form or ground in oil or in japan to form a paste.

I. MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be a mixture of lead chromate and iron ferro and ferricyanide blue, with or without other insoluble compounds of lead, co-precipitated on a base of barium sulfate or insoluble siliceous material or any mixture thereof.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan.

II. PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and tone shall be equal to, and the tinting strength not less than, that of a sample mutually agreed on by buyer and seller.

(*b*) The dry pigment shall meet the following requirements:

	MAXIMUM	MINIMUM
Sum of the barium sulfate and insoluble siliceous material, per cent	80	..
Color (total of insoluble lead compounds and iron blue), per cent	..	20
Percentage of total lead present in the form of chromate	..	75
Total calcium oxide in any form soluble in acid, per cent	0.5	..
Coarse particles (total residue retained on a No. 325 screen), per cent	1.5	..

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances. The paste shall meet the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	88	80
Linseed oil, per cent	20	12
Coarse particles and skins (total residue retained on a No. 325 screen), per cent of the pigment	2.0	..

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard flat coat. It shall meet the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	88	80
Vehicle (japan), per cent	20	12
Coarse particles and skins (total residue retained on a No. 325 screen), per cent	2.0	..
Non-volatile matter in vehicle, per cent of the vehicle	..	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

NOTE.—For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE METHODS OF TESTING SHELLAC VARNISH[1]

Serial Designation: D 214 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Shellac varnishes are made by dissolving shellac in alcohol and are designated in the trade in terms of pounds of shellac per gallon of alcohol, the 4, 4.5 and 5-lb. cut goods being most commonly used. Specially denatured alcohol, Formula No. 1 is largely used, and occasionally wood alcohol and butyl alcohol. Pure grain alcohol, or Formula No. 35 denatured is used for confectioner's varnish or glaze.

DETERMINATION OF BODY (SOLIDS)

APPARATUS

1. The apparatus shall consist of the following:

(*a*) A weighing bottle for volatilized liquids or a Grethan pipette.

(*b*) A tin-foil dish, $1\frac{1}{4}$ in. in height and $2\frac{1}{2}$ in. in diameter.

(*c*) Prepared Sand, prepared as follows: Take sea-sand, sieve it to take out all foreign material. Digest with hot HCl for about one hour. Wash with water to remove all the acid and soluble impurities. Ignite and put in a desiccator to cool. Then put it in a bottle and stopper it tightly.

PROCEDURE

Procedure.

2. (*a*) Put 10 g. of the prepared sand and a short glass rod into the tin-foil dish. Weigh them all together and record the weight accurately. Run into the dish about 1 g. of the sample from the weighing bottle. Record the exact weight of the sample taken into the dish.

(*b*) Mix the varnish and sand with the glass rod.

NOTE.—This is especially important in testing varnish of over 6 lb. so that the varnish and sand will be intimately mixed.

Place in an oven at a temperature of 105° C. and heat for one hour. At the end of one hour take out, cool and weigh. The increase in weight of the tin-foil, sand and glass rod is the weight of the shellac in the varnish.

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

Calculation

3. (*a*) In the case of orange shellac varnish, divide the percentage of the shellac found by 0.98 because orange shellac loses 2 per cent in heating for an hour at 105° C.

(*b*) In the case of white shellac varnish, divide the percentage of shellac found by 0.95 because white shellac varnish is made from bone dry shellac, which contains 5 per cent of moisture.

Example.—

Orange Shellac Varnish:

Weight of varnish taken.............................. 1.3200 g.
Weight of residue (after drying)......................... 0.5491 g.

$$\text{Per cent of body} = \frac{0.5491}{0.98 \times 1.3200} \times 100 = 42.45 \text{ per cent}$$

This percentage corresponds to 5 lb. per gallon.

White Shellac Varnish:

Weight of varnish taken.............................. 1.2973 g.
Weight of residue (after drying)......................... 0.5235 g.

$$\text{Per cent of body} = \frac{0.5235}{0.95 \times 1.2973} \times 100 = 42.45 \text{ per cent}$$

This percentage corresponds to 5 lb. per gallon.

(*c*) To convert the percentage of solids (residue) of the varnish into pounds per gallon, from the percentage of solids expressed in pounds of solids in 100 lb. of varnish, the pounds of solvent is found by difference. The weight of solvent divided by 6.7793 (the weight of one gallon of denatured alcohol (Formula No. 1) at 16° C. (60° F.) gives the number of gallons of alcohol present. From this the number of pounds of shellac per gallon of alcohol is determined.

Example.—

$$100 - 30.68 \text{ (Solids found)} = 69.32 \text{ lb. of alcohol}$$

$$\frac{69.32}{6.7793} = 10.23 \text{ gallons of alcohol.}$$

$$\frac{30.68}{10.23} = 3 \text{ lb. per gallon.}$$

For convenience, tables are appended hereto giving pounds of shellac per gallon for varnishes for various specific gravities.

DETERMINATION OF WAX

4. The wax shall be determined in accordance with the Standard Method of Test for Determination of Wax in Shellac (Serial Designation: D 29) of the American Society for Testing Materials.[1]

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[1] A.S.T.M. Standards Adopted in 1925.

DETERMINATION OF PURITY

5. Purity of varnish of both white and orange shellac variety may be ascertained by determining the iodine number on the gum residue. Two methods are available:

Method 1.—Run 0.5 to 0.7 cc. of varnish into a weighed 6 or 8-oz. glass-stoppered Erlenmeyer flask (a light weight bottle with hollow stopper). Add 1 or 2 cc. of alcohol to spread the varnish film from the bottom of the flask. Place the flask in a hot water bath (90° C.) for 30 minutes and aspirate the alcohol vapor. Cool, weigh and add 20 cc. of acetic acid (melting point 14.8° C.). Proceed in accordance with the method for iodine number described in the Determination of Rosin in the Standard Methods of Testing Shellac (Serial Designation D 29) of the American Society for Testing Materials.[1]

Method 2.—

APPARATUS

A weighing bottle for the adulteration of liquids, or a Grethan pipette.

PROCEDURE

With the percentage of solids known, weigh out enough of the original varnish to correspond to 0.200 g. of solids (0.450 to 0.500 g. of varnish). This is satisfactory in the case of slight adulteration. If heavy adulteration is suspected, weigh out an amount of varnish to correspond to 0.150 g. of solids or less.

From the weighing bottle or pipette weigh, by difference, the amount of varnish selected into a 250-cc. glass-stoppered bottle. Warm in a water bath at not over 78° C. for about 15 minutes and aspirate the alcohol vapor until practically dry. Cool, weigh and add 20 cc. of acetic acid (melting point 14.8° C.). Proceed in accordance with the method for iodine number described in the Determination of Rosin in the Standard Methods of Testing Shellac (Serial Designation: D 29) of the American Society for Testing Materials.[1]

6. *Qualitative Test.*—A qualitative test may be applied to the varnish, and if this shows only adulteration with copal the amount of copal may be approximately determined from the iodine number with the iodine number of copal taken as 130 for purposes of calculation.

In making the qualitative test, filter some of the varnish through dry paper into a large test tube (6 by $\frac{3}{4}$ in.). When 10 cc. has run through, fill the test tube with 99-per-cent methyl alcohol and agitate.

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[1] 1924 Book of A.S.T.M. Standards.

A pure shellac will remain clear and when adultered with copal will give a precipitation on standing.

A qualitative test for rosin may be made by the Halphen-Hicks method as described by Walker and Steele, *Bulletin No. 232*, Bureau of Standards, March, 1923, pp. 284–285.

COMPLETE VARNISH ANALYSIS

7. A complete varnish analysis would include distilling about 50 cc. of varnish to dryness on a sand bath and redistilling the distillate to determine the boiling point. A portion of the final distillate should be diluted with water to ascertain if other solvents, such as naphtha or benzol, have been added. These will separate out and the separated layer may be measured.

APPENDIX

RELATION OF POUNDS OF SHELLAC PER GALLON TO SPECIFIC GRAVITY

WHITE SHELLAC VARNISH: DRY BLEACHED SHELLAC (5 PER CENT MOISTURE), DENATURED ALCOHOL, FORMULA NO 1, 190 PER CENT PROOF

SHELLAC, LB. PER GAL. OF ALCOHOL	SPECIFIC GRAVITY, 16° C. (60° F.)	DEG. BAUMÉ	WEIGHT PER 1 GAL. VARNISH, LB.
3	0.9056	24.60	7.55
3.5	0.9167	22.70	7.64
4	0.9278	20.90	7.74
4.5	0.9375	19.33	7.79
5	0.9464	17.92	7.89
5.5	0.9530	16.90	7.95
6	0.9597	15.90	8.00
ORANGE SHELLAC VARNISH: TN SHELLAC (2 PER CENT MOISTURE), DENATURED ALCOHOL, FORMULA NO. 1, 190 PER CENT PROOF			
3	0.900	25.55	7.50
3.5	0.9114	23.6	7.60
4	0.9228	21.7	7.69
4.5	0.9318	20.25	7.77
5	0.9395	19.0	7.83
5.5	0.9500	17.36	7.92
6	0.9554	16.53	7.97

NOTE 1.—Shellac varnishes will not always agree exactly with the above specific gravity, estimated at 16° C. (60° F.). They will vary somewhat, due to more or less moisture and insoluble matter contained in the white and orange shellacs, and also to loss of solvent by evaporation. However, the above figures agree closely with theoretical and practical results.

NOTE 2.—Ten pounds of shellac cut in alcohol yields a volume of 1 gallon. This must be taken into consideration in determining the weight per gallon of varnish.

TENTATIVE METHODS OF ROUTINE ANALYSIS OF WHITE LINSEED OIL PAINTS[1]

Serial Designation: D 215 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

PRELIMINARY PROCEDURE

On receipt of a sample make a record of the label, noting especially the brand, the name of the manufacturer, and any statement as to composition and net contents. Weigh the unbroken package, open, note odor and condition of the contents, pour into a clean container, and mix thoroughly by pouring from one container to the other, finally leaving the well-mixed sample in the second container, which shall be tightly closed. The well-mixed sample is used at once for the determinations described under "Methods." The original can and cover may be cleaned with gasoline, wiped dry, and then weighed. This weight subtracted from the original weight will give the net weight of the contents. If desired, the specific gravity of the paint may be determined and the weight per gallon calculated, and the volume of paint and the capacity of the container may be measured.

REAGENTS REQUIRED

Extraction Mixture.—10 volumes ether (ethyl ether);
6 volumes benzol;
4 volumes methyl alcohol;
1 volume acetone.

Aqueous Sodium Hydroxide.—Dissolve 100 g. of NaOH in distilled water and dilute to 300 cc.

Alcoholic Sodium Hydroxide Solution.—Dissolve pure NaOH in 95-per-cent ethyl alcohol in the proportion of about 22 g. per 1000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle and keep well stoppered. This solution should be colorless or only slightly yellow when used, and it will keep colorless longer if the alcohol is previously treated with sodium hydroxide (about 80 g. to 1000 cc.), kept at about 50° C. for 15 days, and then distilled.

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

Hanus Solution.—Dissolve 13.2 g. of iodine in 1000 cc. of 99.5-per-cent glacial acetic acid which will not reduce chromic acid. Add enough bromine to double the halogen content as determined by titration (3 cc. of bromine is about the proper amount). The iodine may be dissolved by the aid of heat, but the solution should be cold when the bromine is added.

Standard Sodium Thiosulfate Solution.—Dissolve pure sodium thiosulfate in distilled water (that has been well boiled to free it from carbon dioxide) in the proportion of 24.83 g. crystallized sodium thiosulfate to 1000 cc. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine, pure potassium biiodate, or pure KIO_3 (see Treadwell-Hall, Analytical Chemistry, Vol. 2.) This solution will be approximately 0.1 N, and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly 0.1 N. Preserve in a stock bottle provided with a guard tube filled with soda lime.

Starch Solution.—Stir up 2 to 3 g. of potato starch or 5 g. of soluble starch with 100 cc. of 1-per-cent salicylic acid solution, add 300 to 400 cc. of boiling water, and boil the mixture until the starch is practically dissolved, then dilute to 1 liter.

Potassium Iodide Solution.—Dissolve 150 g. of potassium iodide free from iodate in distilled water and dilute to 1000 cc.

Acid Ammonium Acetate Solution.—Mix 150 cc. of 80-per-cent acetic acid, 100 cc. of water, and 95 cc. of strong ammonia (sp. gr. 0.90).

Ammonium Polysulfide.—Pass H_2S gas into 200 cc. of strong ammonium hydroxide (sp. gr. 0.90) in a bottle immersed in running water or in iced water until the gas is no longer absorbed; then add 200 cc. of strong ammonium hydroxide (sp. gr. 0.90) and dilute with water to 1000 cc. Digest this solution with 25 g. of flowers of sulfur for several hours and filter.

"Lead Acid."—Mix 300 cc. of H_2SO_4 (sp. gr. 1.84) and 1800 cc. of distilled water. Dissolve 1 g. of c. p. lead acetate in 300 cc. of distilled water and add this to the hot solution, stirring meanwhile. Let stand at least 24 hours and siphon through a thick asbestos filter.

Potassium Permanganate Solution.—Dissolve 3.2 g. of pure potassium permanganate in a liter of distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and standardize as follows: In a 400-cc. beaker dissolve 0.40 to 0.50 g. of Bureau of Standards' sodium oxalate in 250 cc. of hot water (80 to 90° C.) and add 15 cc. of dilute sulfuric acid (1:1). Titrate at once with the potassium permanganate solution, stirring the liquid vigorously and continuously. The permanganate must not be added more rapidly than 10 to 15 cc. per minute, and the last 0.5 to 1 cc. must be added dropwise with particular care to allow each drop to be fully decolorized before the next is introduced. The temperature of the solution should not be below 60° C. by the time the end point is reached. (Too rapid cooling may be prevented by allowing the beaker to stand on a small asbestos-covered hot plate during the titration. The use of a small thermometer as a stirring rod is most convenient.) The weight of sodium oxalate used multiplied by 0.8334 gives its iron equivalent. The permanganate solution should be kept in a glass stoppered bottle painted black to keep out light.

The iron (Fe) value of the $KMnO_4$ multiplied by 1.076 theoretically equals its antimony (Sb) equivalent. However, for use in determining antimony, the $KMnO_4$ is best standardized as follows: To 0.25 g. of pure metallic antimony in a 500-cc. Pyrex Erlenmeyer flask, add 12 to 15 cc. of concentrated H_2SO_4 and 10 to 12 g. of K_2SO_4; heat until all the antimony is dissolved, cool, dilute to 250 cc. with water, add 20 cc. of concentrated HCl, cool to 10 to 15° C., and titrate with the $KMnO_4$ solution until a faint pink color is obtained. For special work, after digesting, dilute to 100 cc. with water, add 1 to 2 g. of Na_2SO_3, and boil until all the SO_2 is expelled. This is shown when no blue color is obtained with starch-iodate paper (see below); the volume will be reduced about one-half. Dilute to 250 cc. with water, add 20 cc. of HCl (sp. gr. 1.19), and complete as described.

Standard Potassium Ferrocyanide.—Dissolve 22 g. of the pure salt in water and dilute to 1000 cc. To standardize, transfer about 0.2 g. (accurately weighed) of pure metallic zinc or freshly ignited pure zinc oxide to a 400-cc. beaker. Dissolve in 10 cc. of HCl and 20 cc. of water. Drop in a small piece of litmus paper, add ammonium hydroxide until slightly alkaline, then add HCl until just acid, and then 3 cc. of strong HCl. Dilute to about 250 cc. with hot water and heat nearly to boiling. Run in the ferrocyanide solution slowly from a burette with constant stirring until a drop tested on a white porcelain plate with a drop of the uranyl indicator shows a brown tinge after standing one minute. A blank should be run with the same amounts of reagents and water as in the standardization. The amount of ferrocyanide solution required for the blank should be subtracted from the amounts used in standardization and in titration of the sample. The standardization must be made under the same conditions of temperature, volume, and acidity as obtained when the sample is titrated.

Uranyl Indicator for Zinc Titration.—A 5-per-cent solution of uranyl nitrate in water or a 5-per-cent solution of uranyl acetate in water made slightly acid with acetic acid.

Alkaline Lead Nitrate Solution.—Into 100 cc. of KOH solution (56 g. in 140 cc. of water) pour a saturated solution of lead nitrate (250 g. in 500 cc. of water) until the precipitate ceases to redissolve, stirring constantly while mixing. Let settle, filter through asbestos, and dilute the clear filtrate with an equal volume of water. About 3 volumes of the lead nitrate solution will be required for one of the KOH.

Ammoniacal Cadmium Chloride or Zinc Sulfate Solution.—Dissolve 8 g. of cadmium chloride in 200 cc. of water and add 200 cc. of NH_4OH (sp. gr. 0.90), or, dissolve 200 g. of zinc sulfate in 1080 cc. of water and 920 cc. of NH_4OH (sp. gr. 0.90).

Standard Potassium Iodate Solution.—Dissolve 3.6 g. of KIO_3 and 39 g. of KI in 1000 cc. of water. For general work the theoretical sulfur titer of this solution should be used; for special work, the solution may be standardized against like material, such as a lithopone of known sulfide sulfur content. The theoretical titer is based on standard $Na_2C_2O_4$ and is obtained as follows: To 300 cc. of water in a 600-cc. flask, preferably glass stoppered, add 10 cc. of concentrated HCl (sp. gr. 1.19) and 1 g. of KI. Cool and add 10 cc. of 0.1 *N* $KMnO_4$ solution which has been standardized against $Na_2C_2O_4$. Swirl gently, stopper, and let stand for five minutes. Titrate the liberated iodine with standard $Na_2S_2O_3$ solution until the color fades. Then add 10 cc. of starch solution and continue the titration until the blue color is destroyed. Repeat the titration with the sole difference that 10 cc. of the iodate solution is substituted for the $KMnO_4$ solution. Calculate the normality of the iodate solution.

Starch Indicator for Sulfur Titration.—(1) To 1000 cc. of boiling water, add a cold suspension of 6 g. of starch in 100 cc. of water and boil vigorously for five minutes. Cool the solution, add 6 g. of $ZnCl_2$ dissolved in 50 cc. of cold water, thoroughly mix and set aside for 24 hours. Decant the clear supernatant liquid into a suitable container, add 3 g. of KI, and mix thoroughly. (2, Optional.) Prepare an emulsion of 6 g. of soluble starch in 25 cc. of water, add a solution of 1 g. of NaOH in 10 cc. of water, and stir the solution until it gelatinizes. Dilute to 1000 cc. with water, add 3 g. of KI, and mix thoroughly.

Starch-Iodate Paper.—Impregnate filter paper with a solution obtained by heating 2 g. of starch with 100 cc. of water, and, after solution, adding 0.2 g. of KIO_3 dissolved in 5 cc. of water.

Standard Iodine Solution for SO_2.—Place 15 to 20 g. of pure KI in a liter flask, dissolve in as little water as possible, and then add about 6.4 g. of resublimed iodine. Shake until the iodine is all dissolved, dilute to the mark with water, and mix. This solution is approximately 0.05 *N* and is standardized against 0.05 *N* $Na_2S_2O_3$ to obtain its true normality.

Standard Sodium Thiosulfate Solution for SO_2.—Prepare and standardize as described above, except that 12.42 g. of pure crystallized $Na_2S_2O_3 \cdot 5H_2O$ are used or the 0.1 *N* solution may be diluted with an equal volume of cold CO_2-free water.

METHODS

WATER (NOTE 1)

Mix 100 g. of the paint in a 250-cc. flask with 75 cc. of toluene. Place the flask in an oil bath, connect with condenser, apply heat to the bath, and distil until about 50 cc. of distillate have been collected in a graduate. The temperature in the flask should be then about 105 to 110° C. The number of cubic centimeters of water collected under the toluene in the receiver is the percentage of water in the paint.

VOLATILE THINNER

Weigh accurately from 3 to 5 g. of the paint into a tared flat-bottomed dish about 8 cm. in diameter, spreading the paint over the bottom. Heat at 105 to 110° C. for one hour, cool, and weigh. Calculate the loss in weight as percentage of water and volatile thinner, subtract from this the percentage of water (1), and report the remainder as volatile thinner.

NATURE OF THE THINNER

Transfer about 150 g. of the paint to a 500-cc. flask fitted with a 2-hole cork stopper carrying a spray trap connected with a vertical condenser. Through the other hole in the stopper pass an influx tube for steam. (This tube should dip below the surface of the paint.) Heat the flask in an oil bath or an air bath at 100° C. and pass through it a current of steam; with the steam still passing through, raise the temperature of the bath to 130° C. Catch the distillate in a small separatory funnel; continue distillation until 300 cc. of water has been condensed. Portions of this water may be drawn from the cock of the separatory funnel from time to time, but care must be taken not to draw out any of the volatile thinner. Let the distillate stand until it separates into two layers, then draw off the water, and filter the volatile thinner through a dry filter paper into a dry flask. If the thinner is apparently turpentine, examine the distillate by the methods described under the Standard Specifications for Turpentine (Serial Designation: D 13) of the American Society for Testing Materials.[1] If the thinner is a mixture of turpentine and mineral spirits, an approximate determination of the amount of turpentine may be made by the polymerization test specified for under turpentine. It should be noted that turpentine is slightly soluble in water (about 0.3 to 0.4 cc. per 100 cc. of water).

To test for benzol, add a few drops of the distillate to a small quantity of a mixture of concentrated HNO_3 and concentrated H_2SO_4, and heat cautiously. The characteristic odor of nitrobenzol will be noted if benzol is present.

If the thinner is apparently all mineral spirits, no further examination is necessary.

PERCENTAGE OF PIGMENT

Strain a portion of the well-mixed sample through a No. 80 sieve to remove any skins and weigh accurately about 15 g. of the strained paint in a weighed centrifuge tube. Add 20 to 30 cc. of "extraction mixture" (see "Reagents"), mix thoroughly with a glass rod, wash the rod with more of the extraction mixture, and add enough of the reagent to make a total of 60 cc. in the tube. Place the tube in the container of a centrifuge, surround the tube with water, and counterbalance the container of the opposite arm with a similar tube, or a tube with water. Whirl at a moderate speed until well settled. Decant the clear supernatant liquid, repeat the extraction twice with 40 cc. of extraction mixture and once with 40 cc. of ethyl ether. After drawing off the ether, set the tube in a beaker of water at about 80° C. or on top of a warm oven for 10 minutes, then in an oven at 105 to 110° C. for two hours. Cool, weigh, and calculate the percentage of pigment. Grind the pigment to a fine powder, pass through a No. 80 sieve to remove any skins, and preserve in a stoppered bottle.

PERCENTAGE OF NON-VOLATILE VEHICLE

Add together the percentages of water, of volatile thinner, and of pigment, and subtract the sum from 100. Report the remainder as non-volatile vehicle.

TESTING NON-VOLATILE VEHICLE

(*a*) *Preparation of fatty acids.*

To about 25 g. of the paint in a porcelain casserole, add 15 cc. of aqueous sodium hydroxide (see "Reagents") and 75 cc. of ethyl alcohol, mix and heat uncovered on a steam bath until all volatile thinner is driven off and saponification is complete. Add 100 cc. of water, boil, add H_2SO_4 (sp. gr. 1.2) (8 to 10 cc. in excess), boil, stir, and transfer to a separatory funnel to which some water has been previously added. Draw off as much as possible of the acid aqueous layer and any insoluble or precipitated matter, wash once with water, then add 50 cc. of water and 50 cc. of ethyl ether. Shake very gently with a whirling action to dissolve the fatty acids in the ether, but not so violently as to form an emulsion. Draw off the aqueous layer and wash the ether layer with one 15-cc. portion of water and then with 5 cc. portions of water until free from sulfuric acid. Then draw off the water layer completely. Transfer the ether solution to a dry flask and add 25 to 50 g. of anhydrous sodium sulfate. Stopper the flask and let stand with occasional shaking at a temperature below 25° C. until the water is completely removed from the ether solution, which will be shown by the solution becoming perfectly clear above the solid sodium sulfate. Decant this clear solution, if necessary, through a dry filter paper into a dry 100-cc. Erlenmeyer flask. Pass a rapid current of dry air (pass through a $CaCl_2$ tower) into the mouth of the Erlenmeyer flask and heat to a temperature below 75° C. on a dry hot plate until the ether is entirely driven off. It is important to follow all of the details, since ether generally contains alcohol, and after washing with water always contains water. It is very difficult to remove water and alcohol by evaporation from fatty acids, but the washing of the ether solution and subsequent drying with anhydrous sodium sulfate removes both water and alcohol. Ether, in the absence of water and alcohol, is easily removed from fatty acids by gentle heat. If the pigment settles out rapidly in a sample of the paint on standing so that sufficient vehicle can be poured off; or, if sufficient vehicle is obtained by centrifuging the paint, it will be advantageous to saponify this separated vehicle and liberate and prepare the fatty acids as described.

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[1] 1924 Book of A.S.T.M. Standards.

The fatty acids prepared as above should be kept in a stoppered flask and examined at once.

(*b*) *Test for mineral oil and other unsaponifiable matter.*

Place 10 drops of the fatty acids Method (*a*), in a 50-cc. test tube, add 5 cc. of alcoholic soda (see "Reagents"), boil vigorously for five minutes, add 40 cc. of water, and mix; a clear solution indicates that not more than traces of unsaponifiable matter are present.

(*c*) *Iodine number of fatty acids* (Note 2).

Place a small quantity of the fatty acids Method (*a*), in a small weighing burette or beaker. Weigh accurately. Transfer by dropping about 0.15 g. (0.10 to 0.20 g.) into a 500-cc. bottle having a well ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of sample used. (If desired the sample may be weighed in a small wide-mouthed vial and the vial containing the weighed sample placed in the bottle or flask.) Add 10 cc. of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc. of chloroform to two empty bottles like that used for the sample. Add to each bottle 25 cc. of the Hanus solution (see "Reagents") and let stand with occasional shaking for 30 minutes. Add 10 cc. of the 15-per-cent potassium iodide solution and 100 cc. of water, and titrate with standard sodium thiosulfate solution (see "Reagents"), using starch as indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titrations and the titration on the sample and the iodine value of the thiosulfate solution, calculate the iodine number of the sample tested. (Iodine number is centigrams of iodine to 1 g. of sample.)

ANALYSIS OF PIGMENT

(a) *Qualitative Analysis.*

A complete qualitative analysis, following the well-established methods, should be made and the quantitative scheme modified as required. Add acetic acid slowly to the pigment until all carbonate is decomposed (noting whether any hydrogen sulfide is evolved); then add a large excess of acid ammonium acetate solution (see "Reagents"), boil, filter, and test the filtrate for metals other than lead and zinc (especially calcium and barium). The absence of calcium in this filtrate indicates that the extending pigments contain no calcium carbonate or calcium sulfate; the absence of barium indicates that the extending pigments contain no barium carbonate (Note 5). Wash the matter insoluble in acid ammonium acetate solution with another portion of this solution, and finally with hot water. This insoluble matter is dried, ignited, and tested for siliceous matter, barium sulfate, and titanium compounds. To test for the latter, place a small amount of the insoluble matter, or of the original sample (about 0.5 g.), in a 250-cc. Pyrex glass beaker; add 20 cc. of concentrated H_2SO_4 and 7 to 8 g. of ammonium sulfate. Mix well, and boil for a few minutes. A residue denotes the presence of silica or siliceous matter. Cool the solution, dilute with 100 cc. of water, heat to boiling, settle, filter, wash with hot 5-per-cent sulfuric acid until free from titanium. The residue may be tested for lead, barium, and silica.

Add hydrogen peroxide to a small portion of the filtrate; a clear yellow-orange color indicates the presence of titanium. Boil another portion of the filtrate with metallic tin or zinc; a pale blue to violet coloration indicates titanium. Treat another portion (about 1 g.) of the pigment with 20 cc. of HCl (1:1) and note whether any H_2S is evolved; boil the solution for about 5 minutes, add about 25 cc. of hot water, filter, and wash with hot water. Render a small portion of the filtrate alkaline with NH_4OH, acidify with HCl, and add a little $BaCl_2$ solution; a white precipitate ($BaSO_4$) indicates the presence of a soluble sulfate. To another portion of the filtrate add a little H_2SO_4; a white precipitate indicates the presence of lead, soluble barium or both (some $CaSO_4$ may also separate); filter, wash to remove free acid, and treat the precipitate with a few drops of KI solution; the formation of yellow PbI_2 indicates the presence of lead. The white precipitate may also be treated with H_2S water; the formation of black PbS indicates the presence of lead. To another portion of the original filtrate add NH_4OH until alkaline, render slightly acid with acetic acid, heat to boiling, and add a little $K_2Cr_2O_7$ solution; a yellow or orange-yellow precipitate indicates the presence of lead, soluble barium or both. To another portion of the original filtrate add a few drops of $K_4Fe(CN)_6$ solution; a white precipitate with a blueish tinge indicates the presence of zinc. Pass into the remaining portion of the original filtrate a current of H_2S for 5 to 10 minutes, add an equal volume of water and pass H_2S into the solution for about 5 minutes; filter, wash with H_2S water; then digest the precipitate with ammonium polysulfide, filter, acidify the filtrate with HCl and warm; the presence of antimony is indicated by the separation of an orange colored precipitate. The filtrate from the H_2S precipitate may be tested for barium, calcium, and magnesium in the usual manner.

(*b*) *Quantitative Analysis.*

(1) *Single Pigments:*

If the sample is a single pigment, follow the method described in the Standard Methods of Routine Analysis of White Pigments (Serial Designation: D 34) of the American Society for Testing Materials[1] for the particular pigment in hand.

(2) *Mixed or Composite Pigments:*

Moisture (Note 3) (*Matter Volatile at 105–110° C.*).—Place 1 to 2 g. of the sample in a wide-mouth, short weighing tube provided with glass stopper. Heat with the stopper removed for 2 hours at a temperature between 105 and 110° C. Insert the stopper, cool, and weigh. Calculate the loss in weight as moisture (matter volatile at 105 to 110° C.).

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[1] 1924 Book of A.S.T.M. Standards.

Loss on Ignition.—Ignite 1 g. of the pigment in a porcelain crucible over a Meker burner to constant weight (Note 4).

Insoluble Matter.—Moisten 1 g. of the sample with a few drops of alcohol, cover, add 40 cc. of HCl (1:1), boil gently for 5 to 10 minutes. Wash off cover, evaporate to dryness, and heat at about 150° C. for one-half to one hour to dehydrate the residue. Moisten the residue with 4 cc. of concentrated HCl, allow to stand a few minutes, dilute with 100 cc. hot water, boil a few minutes, filter hot through paper, wash with hot water (till washings give no test for lead and chlorine). Ignite the paper and residue in a platinum or porcelain crucible, cool, and weigh total insoluble matter (Note 5). (The insoluble matter may be filtered off on a Gooch crucible, washed with hot water, dried at 105° C., cooled, and weighed; then ignited, cooled, and weighed, when it is desired to get the loss on ignition (combined water, organic matter, etc.) of same, or the insoluble matter is not to be further examined.) If the sample contains titanium pigment, practically all of the TiO_2 will be found in the insoluble matter along with $BaSO_4$ and siliceous matter. The TiO_2 may be determined in the insoluble matter or in a separate portion of the original sample by the method described in the Standard Methods of Routine Analysis of Titanium Pigments (Serial Designation: D 186) of the American Society for Testing Materials.[1] To determine $BaSO_4$, mix the ignited insoluble matter with about 10 times its weight of anhydrous sodium carbonate (grinding the mixture in an agate mortar if necessary), and fuse the mixture in a covered platinum crucible, heating about one hour. Let cool, place crucible and cover in a 250-cc. beaker, add about 100 cc. of water, and heat until the melt is disintegrated. Filter on paper (leaving crucible and cover in beaker) and wash the beaker and filter thoroughly with hot water to remove soluble sulfates. Place the beaker containing the crucible and cover under the funnel, pierce the filter with a glass rod, and wash the residue into the beaker by means of a jet of hot water. Wash the paper with hot dilute HCl (1:1) and then with hot water. Remove crucible and cover, washing them with a jet of hot water and removing any adhering precipitate. Add cautiously 20 cc. of concentrated H_2SO_4 and evaporate until fumes of H_2SO_4 are evolved and the precipitated matter is dissolved. *Cool,* add cautiously, with stirring, about 100 cc. of water, and boil a few minutes. Let the precipitate settle, filter on a weighed Gooch crucible, wash with hot water, ignite, cool, and weigh as $BaSO_4$. Subtract the sum of the percentages of $BaSO_4$ and TiO_2 from the percentage of total insoluble matter and report the result as the percentage of insoluble siliceous matter (Note 6).

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[1] A.S.T.M. Standards Adopted in 1925.

If it is desired to examine the siliceous matter, unite the filtrates from the Na_2CO_3 fusion and the $BaSO_4$ precipitate, acidify with HCl, evaporate to dryness, and proceed as in a silicate analysis, taking cognizance of any TiO_2 that may be found, if titanium pigment were originally present.

Total Lead (Antimony).—Unite the filtrate and washings (total volume 150 to 200 cc.) from the total insoluble matter, pass H_2S into the solution until it is saturated, add an equal volume of water, and again saturate with H_2S. Filter, wash with water containing a little hydrogen sulfide, dissolve in hot HNO_3 (1:3), washing the paper with hot water; add 10 to 20 cc. of H_2SO_4 (1:1), evaporate until copious fumes of sulfuric acid are evolved; cool, add about 75 cc. of water, and then about 75 cc. of 95-per-cent ethyl alcohol. Stir, let settle, filter on a Gooch crucible, wash with dilute alcohol, dry in an oven at 105 to 110° C.; or, ignite gently in a radiator[1] or muffle, cool, and weigh as $PbSO_4$. Calculate to PbO (Note 7).

If the pigment contains antimony, filter and wash the sulfide precipitate as above; then wash the precipitate with a fine jet of H_2O from the paper into a porcelain dish or casserole, add 25 cc. of ammonium polysulfide (see "Reagents"), cover the vessel, and warm the mixture at 40 to 60° C. for 10 to 15 minutes with frequent stirring. Wash off cover, filter through the paper used in the first case, and wash with 2 to 3-per-cent Na_2S or $(NH_4)_2S$ solution. Discard the filtrate. Dissolve the residue in hot dilute HNO_3 (1:3), and determine the lead as $PbSO_4$, as described above. Or, the original sulfide precipitate may be discarded and the lead determined on a separate portion of the pigment as follows: To 1 g. of the sample in a covered beaker, add 40 cc. of HCl (1:1) and boil gently for 5 to 10 minutes. Wash off cover and evaporate to dryness. Moisten the residue with a few drops of HCl, add about 50 cc. of hot water, boil a few minutes, filter hot through paper, and wash with hot water until washings give no test for lead. (If the sample contains no insoluble matter, the filtration is omitted.) To the filtrate add 20 cc. of H_2SO_4 (sp. gr. 1.84) and evaporate until dense white fumes of H_2SO_4 are copiously evolved. Allow to cool, but not below 60° C., and then add slowly 50 cc. of water while the solution is agitated. Heat to boiling for several minutes in order to insure complete solution of antimony sulfate. Allow the $PbSO_4$ to settle out until the supernatant liquid is clear, not letting the temperature fall below 60° C. If the liquid does not clear quickly it must be heated longer. When clear, pour the solution through a weighed porcelain Gooch crucible with asbestos mat, decanting the solution as completely as possible without allowing more than a very small amount of $PbSO_4$ to go over into the crucible. Now add 10 cc. more of H_2SO_4 (sp. gr. 1.84) to the $PbSO_4$ in the original beaker, and boil for several minutes. Cool, add slowly 30 cc. of water, and again heat to boiling for a few minutes; allow the solution to cool to about 60° C. and completely transfer the $PbSO_4$ to the Gooch crucible. Wash with "lead acid" (see "Reagents") to remove soluble sulfates and finally wash free of acid with dilute alcohol (equal parts of ethyl alcohol or denatured alcohol and water). Dry in an oven at 105 to 110° C., or, ignite gently in a radiator or muffle. Calculate to PbO. Or, determine as chromate as described below.

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[1] *U. S. Geological Survey Bulletin 700*, p. 33 (1919).

If soluble compounds of barium or calcium are present, $BaSO_4$ and $CaSO_4$ will be included with the $PbSO_4$. If soluble SiO_2 is present, it will also be included with the $PbSO_4$. In such cases, the $PbSO_4$ precipitate after washing with dilute alcohol may be dissolved in acid ammonium acetate (see "Reagents") and the lead determined as $PbCrO_4$, as described below. For ordinary work, the amount of $BaSO_4$ dissolved by the acetate treatment may be disregarded.

If the pigment contains no soluble antimony, barium, or calcium compounds, the lead may be determined directly on the original pigment, as follows: To 1 g. of the sample in a covered beaker, add 25 cc. of HNO_3 (1:1), and boil gently a few minutes. Wash off cover, evaporate to dryness on a steam bath, moisten with HNO_3, add hot water, and heat a few minutes. Filter and wash with hot water until washings are lead-free. Add 10 to 20 cc. of H_2SO_4 (1:1) to the clear solution, evaporate and determine lead as $PbSO_4$, as above described.

In the absence of soluble compounds of antimony, iron, aluminum, and barium, the following procedure may be used: Treat 1 g. of the original pigment with 25 cc. of HNO_3 (1:1) and proceed as above. To the clear solution, diluted to 200 cc. add NH_4OH in slight excess, acidify with acetic acid, and add 4 to 6 cc. more of this acid; heat to boiling and add 10 to 15 cc. of a 10-per-cent solution of $K_2Cr_2O_7$. Heat until the yellow precipitate assumes an orange color, let settle and filter on a weighed Gooch crucible, wash by decantation until the washings are colorless, finally transferring all of the precipitate. Then wash with 95-per-cent ethyl alcohol and then with ether; dry to constant weight at 110° C., cool, and weigh $PbCrO_4$. Calculate to PbO.

Antimony Oxide.—Transfer 0.3 g. of a straight antimony oxide pigment, or 0.5 g. of a mixed pigment, to a 500-cc. Pyrex Erlenmeyer flask, add 15 cc. of water and 25 cc. of concentrated HCl (sp. gr. 1.19). Cover with a watch glass, warm on the steam bath 10 to 15 minutes to dissolve the antimony oxide, wash off cover, add 250 cc. of water, and 15 cc. of concentrated H_2SO_4 (sp. gr. 1.84). Boil 2 minutes, cool to 10 to 15° C., and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution (see "Reagents"). Calculate to Sb_2O_3.

The above procedure gives only the antimony in the *ous* condition. The following method gives the total antimony (*ous* and *ic* forms): Transfer 0.3 g. of a straight antimony oxide pigment, or 0.5 g. of a mixed pigment, to a 500-cc. Pyrex Erlenmeyer flask, add 15 cc. of H_2SO_4 (sp. gr. 1.84), 10 g. of K_2SO_4, and a 9-cm. filter paper (to furnish carbon to act as a reducing agent). Place a funnel in the neck of the flask, and heat until the solution becomes colorless. Cool, wash off the funnel, dilute to 250 cc. with water, add 20 cc. of concentrated HCl, and boil 2 minutes; cool to 10 to 15° C., and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution. For special work, after digesting, dilute to 100 cc. with water, add 1 to 2 g. of Na_2SO_3, and boil until all the SO_2 is expelled. This is shown when no blue color is obtained with starch-iodate paper (see "Reagents"); the volume will be reduced about one-half. Dilute to 250 cc. with water, add 20 cc. of HCl (sp. gr. 1.19), and complete the determination as described. Calculate total Sb to Sb_2O_3.

Antimony Oxide (in the presence of appreciable amounts of iron).—Treat 1 g. of the mixed pigment, or 0.3 g. of a straight antimony oxide pigment, in a covered 250-cc. beaker with 5 cc. of water and 20 cc. of HCl (sp. gr. 1.19); heat on the steam bath for 15 minutes, cool, wash off cover, add 3 g. of tartaric acid and 100 cc. of hot water, and digest a few minutes. Filter, catching the filtrate in a 500-cc. Pyrex Erlenmeyer flask; wash thoroughly with hot water, dilute to 300 cc. with hot water, and pass in H_2S until the precipitation is complete. (If the sample contains no insoluble matter, disolve directly in a 500-cc. Pyrex Erlenmeyer flask, add tartaric acid, dilute, and pass in H_2S.) Filter, wash with water containing H_2S until free from HCl, return paper and precipitate to the Erlenmeyer flask, add 15 cc. of H_2SO_4 (sp. gr. 1.84) and 10 g. of K_2SO_4, place a funnel in the neck of the flask, and heat until the solution is colorless. Cool, wash off the funnel, carefully dilute the solution to 100 cc. with water, add about 1 g. of Na_2SO_3, and boil until all of the SO_2 is expelled. (Solution will be reduced to less than one-half its former volume.) This is shown when no blue color is obtained with starch-iodate paper (see "Reagents"). Dilute to about 250 cc. with water, add 20 cc. of HCl (sp. gr. 1.19), boil for 2 or 3 minutes, cool to about 10° C., and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution (see "Reagents"). Calculate the total antimony to Sb_2O_3.

Soluble Barium.—Boil the combined filtrate and washings, reduced in volume by evaporation if need be, from the PbS precipitate (Total Lead) to expel H_2S. Add a slight excess of H_2SO_4 (1:4) over the amount required to precipitate the barium, heat to boiling, let stand on a steam bath about one hour, filter on a weighed Gooch crucible, wash with hot water, dry, ignite, cool, and weigh $BaSO_4$ (Notes 5 and 8). Calculate to BaO.

Alumina (Fe_2O_3, TiO_2, P_2O_5).—Boil the filtrate from the PbS to expel H_2S, add a few drops of HNO_3, and continue the boiling a few minutes to oxidize any iron that may be present. In case soluble barium was present, use the filtrate from that determination. To the solution containing at least 5 g. of NH_4Cl per 200 cc. of solution, or an equivalent amount of HCl, add a few drops of methyl red (0.2-per-cent alcoholic solution) and heat just to boiling. Carefully add dilute NH_4OH drop by drop until the color of the solution changes to a distinct yellow. Boil the solution for one to two minutes and filter at once. Wash the precipitate thoroughly with hot 2-per-cent NH_4Cl solution (Note 9). Ignite the precipitate, cool, and weigh as Al_2O_3 (Note 10).

Total Zinc.—(*a*) To the combined filtrate and washings from the alumina precipitate, add sufficient NH_4Cl to give 5 g. per 100 cc. of solution, and then add 1 g. of ammonium acetate.[1]

Render slightly acid with acetic acid and pass in a current of H_2S to saturation. Allow the precipitate to settle completely, filter on paper, and wash with a 2-per-cent solution of acetic acid saturated with H_2S. Transfer the precipitate and filter to the vessel in which the precipitation was effected, add 30 cc. of water and 10 cc. of concentrated HCl, heat until all zinc is in solution, add 200 cc. of water and a small piece of litmus paper; add strong NH_4OH until slightly alkaline, render just acid with HCl, then add 3 cc. of concentrated HCl, heat nearly to boiling, and titrate with standard potassium ferrocyanide solution as in standardizing that solution (see "Reagents").

(*b*) Zinc may be determined directly on the original sample as follows (Note 11): Weigh accurately about 1 g. (or an amount that will give a burette reading approximately equal to that obtained in the standardization) of the pigment, transfer to a 400-cc. beaker, add 30 cc. of HCl (1:2), boil a few minutes, add 200 cc. of water and a small piece of litmus paper; add strong NH_4OH until slightly alkaline, render just acid with HCl, then add 3 cc. of concentrated HCl, heat nearly to boiling, and titrate with standard $K_4Fe(CN)_6$ solution as in standardizing that solution (see "Reagents").

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[1] F. A. Gooch, "Representative Procedures in Quantitative Chemical Analysis," 1st Ed., p. 107.

(*c*) When iron is present, total zinc may be determined directly on the original sample as follows (Note 11): Weigh accurately about 1 g. (or an amount that will give a burette reading approximately equal to that obtained in the standardization) of the pigment, transfer to a 250-cc. beaker, moisten with alcohol, add 30 cc. of HCl (1:2), boil for 2 or 3 minutes, and add about 100 cc. of water. Add about 2 g. of NH_4Cl, make slightly alkaline with NH_4OH, heat to boiling, let settle on steam bath, filter into a 400-cc. beaker and wash the residue once with hot water. Remove the 400-cc. beaker and pour dilute HCl on the residue, catching the filtrate therefrom in the 250-cc. beaker, wash a few times with hot water. Add to this filtrate 1 g. of NH_4Cl and make slightly alkaline with NH_4OH, boil, let settle, filter on paper used for first filtration, and wash thoroughly with hot water, catching the filtrate and washings in the 400-cc. beaker containing the first filtrate. Add a small piece of litmus paper, acidify with HCl, add 3 cc. of concentrated HCl, heat nearly to boiling, and titrate with standard $K_4Fe(CN)_6$ as above.

(*d*) With pigments containing ZnO and ZnS the ZnO may be determined as follows: Weigh accurately 2.5 g. of the pigment, transfer to a 250-cc. graduated flask, moisten with a few drops of alcohol, add about 200 cc. of 2 to 3-per-cent acetic acid, shake vigorously and let stand for 30 minutes at room temperature, shaking once every 5 minutes. Then let stand at room temperature at least 5 hours, preferably overnight. Fill to the mark with 2 to 3-per-cent acetic acid, mix, filter through a dry paper, discard the first 25 cc. and transfer 100 cc. of the filtrate (corresponding to 1 g.) to a 400-cc. beaker. To the clear solution add 30 cc. of HCl (1:2), 100 cc. of H_2O, and a small piece of litmus paper; add strong NH_4OH until slightly alkaline, render just acid with HCl, then add 3 cc. of concentrated HCl, heat nearly to boiling, and titrate with $K_4Fe(CN)_6$ as above. Calculate the percentage of ZnO (any $ZnCO_3$ or $ZnSO_4$ is included in the ZnO). Subtract this result from the percentage of total Zn as ZnO, and calculate the difference to ZnS.

Soluble Calcium.—Heat the united filtrate and washings, reduced in volume if need be, from the ZnS precipitate, to boiling, add 1 cc. of NH_4OH and an excess of a hot saturated ammonium oxalate solution. Continue the boiling until the precipitate becomes granular; let stand about one hour, filter, and wash with hot water. Ignite, cool, and weigh as CaO (Notes 5, 12, 13); or, place the beaker in which the precipitation was made under the funnel, pierce the apex of the filter with a stirring rod and wash the precipitate into the beaker with hot water, pour warm dilute H_2SO_4 (1:4) through the paper and wash a few times. Add about 30 cc. of dilute H_2SO_4 (1:4), dilute to about 250 cc. heat to 90° C. and titrate at once with standard (0.1 *N*) $KMnO_4$ solution (the temperature of the solution should not be below 60° C. when the end point is reached. See "Reagents"). Calculate to CaO (Notes 5, 12, 13). (The Fe value of $KMnO_4 \times 0.502 =$ CaO value.)

Soluble Magnesium.—Acidify the filtrate from the calcium precipitate with HCl, add 10 cc. of a saturated solution of $Na(NH_4)HPO_4$ and NH_4OH drop by drop, with constant stirring. When the crystallin $(NH_4)MgPO_4$ has formed, add 5 cc. excess of NH_4OH. Allow the solution to stand in a cool place for not less than 4 hours, preferably overnight (Note 14); filter and wash with water containing 2.5 per cent of NH_3. Dissolve the precipitate in a small quantity of hot dilute HCl, dilute the solution to about 100 cc. with water, add 1 cc. of a saturated solution of $Na(NH_4)HPO_4$ and NH_4OH drop by drop, with constant stirring, until the precipitate is again formed as described, and then add 5 cc. excess of NH_4OH. Let the precipitate stand in a cool place for not less than 2 hours, filter on a Gooch crucible, wash with water containing 2.5 per cent of NH_3, ignite, cool, and weigh as $Mg_2P_2O_7$ (Note 15). Calculate to MgO.

Carbon Dioxide.—Determine by evolution with dilute acid and absorption in soda lime or KOH solution. The method given in U. S. Geological Survey Bulletin 700, p. 218, shows a convenient apparatus for carrying out this determination. Use from 1 to 2 g. of the pigment, depending upon the probable CO_2 content, following the method for the determination of carbon dioxide in lime described in the Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 - 25 T) of the American Society for Testing Materials[1] (Note 16).

Total Soluble Sulfur Compounds (Note 5).—Treat 1 g. of the pigment in a 400-cc. beaker with 10 cc. of H_2O, 10 cc. of strong HCl saturated with bromine, and 5 g. of NH_4Cl, digest (covered) on a steam bath for 5 minutes, dilute with hot water to about 200 cc., boil for 5 minutes, filter to separate any insoluble matter, and wash thoroughly with hot water. Nearly neutralize the clear solution in a covered beaker with NaOH solution, complete the neutralization with dry Na_2CO_3 and add about 2 g. more of this reagent. Boil 10 to 15 minutes, wash off cover, let settle, filter, and wash with hot water. Re-dissolve the precipitate in HCl (1:1), reprecipitate with Na_2CO_3 as above, filter, and wash thoroughly with hot water. Acidify the united filtrates with HCl, adding about 1 cc. in excess. Boil to expel bromine, and to the clear boiling solution add slowly with stirring an excess of a 10-per-cent $BaCl_2$ solution. Let stand on a steam bath for at least 1 hour, filter on a weighed Gooch crucible, wash thoroughly with boiling water, dry, ignite at a dull red heat, cool, and weigh as $BaSO_4$. This will include soluble sulfates, SO_3 formed from SO_2 and the SO_3 that is formed from sulfide sulfur (Note 8).

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[1] See p. 618.

Soluble Sulfate (Note 5).—Treat 1 g. of the pigment with 10 cc. of H_2O and 10 cc. of concentrated HCl and 5 g. of NH_4Cl. Boil until H_2S is expelled, adding more HCl (1:1) if necessary; dilute with hot water to about 200 cc., boil for 5 minutes, filter to separate any insoluble matter, and wash thoroughly with hot water. Nearly neutralize the clear solution with NaOH solution and make a double precipitation with Na_2CO_3, as in preceding method, finally weighing as $BaSO_4$, as described above (Note 8).

Sulfide Sulfur[1] (Note 17).—Place 0.5 to 1 g. of the pigment in a flask with about 10 g. of "feathered" or mossy zinc, add 50 cc. of water; insert a stopper carrying a separatory funnel and an exit tube. Run in 50 cc. of concentrated HCl from the funnel, having previously connected the exit tube to two absorption flasks in series; the first flask contains 100 cc. of alkaline lead-nitrate solution (see "Reagents"), the second flask, 50 cc. of the same solution as a safety device. After all of the acid has run into the evolution flask, heat slowly, finally boiling until the first appearance of steam in the first absorption flask. Disconnect, let the lead sulfide settle, filter, wash with cold water, then with hot water till neutral to litmus paper and washings give no test for lead. Dissolve the PbS precipitate in hot, dilute HNO_3, and determine the lead as $PbSO_4$. Calculate to S. For very rapid work, the evolved H_2S may be absorbed in an ammoniacal $CdCl_2$ or $ZnSO_4$ solution (see "Reagents") contained in 2 flasks connected in series, the contents of the absorption flasks washed into a vessel with cold water and diluted to about one liter, acidified with concentrated HCl and titrated with standard potassium iodate solution (see "Reagents"), using starch indicator (see "Reagents").

Sulfur Dioxide (Note 18).—Transfer 10 g. of the pigment to a suitable flask, insert a stopper fitted with a separatory funnel and a spray trap delivery tube (Note 19), and attach the latter to a condenser. Place about 150 cc. of HCl (1:3) in the funnel, the stopcock being closed (Note 20), connect the other end of the condenser with a delivery tube which passes through a 2-hole stopper and extends nearly to the bottom of an absorption flask; through the other hole of the stopper connect a tube or flask to serve as a safety device. Place 25 cc. of 0.05 *N* iodine solution (see "Reagents") in the absorption flask (dilute with water if need be) and 20 cc. of 10-per-cent KI solution in the safety tube; fit stopperin the absorption flask. Open the stopcock and allow the acid to slowly enter the flask. Before all of the acid is admitted, air (washed with NaOH solution) is forced through the top of the separatory funnel (about 2 bubbles per second in the KI solution). Boil the solution 3 minutes with the air passing through, then remove the source of heat and pass air through for 30 minutes. Disconnect the absorption vessels, wash the KI solution into the iodine solution, and titrate at once with 0.05 *N* $Na_2S_2O_3$ solution, using starch indicator. Run a blank determination in exactly the same manner except for the omission of the pigment. Subtract this figure from the previous one and calculate the final result to SO_2 (1 cc. 0.05 *N* I = 0.0016 g. SO_2).

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[1] Evolution Method of W. G. Scott, "White Paints and Painting Materials," p. 257; see also Blair, "The Chemical Analysis of Iron."

Matter Soluble in Water.—Transfer 2.5 g. of the pigment to a graduated 250-cc. flask, add 100 cc. of water, boil for 5 minutes, cool, fill to mark with water, mix, and allow to settle. Pour the supernatant liquid through a dry filter paper and discard the first 20 cc. Then evaporate 100 cc. of the clear filtrate to dryness in a weighed dish, heat for one hour at 105 to 110° C., cool, and weigh. Calculate the percentage. The nature of this may be determined by further examination, as the percentages of SO_3 and CaO may be indicative.

NOTES

1. A convenient apparatus for this determination is shown in Fig. 1 (*b*) of the Standard Method of Test for Water in Bituminous Materials (Serial Designation: D 95) of the American Society for Testing Materials.[1]

2. In the absence of much gum or resin the iodine number gives the best, though doubtful, indication of linseed oil. The fatty acids may be tested for rosin and examined for unsaponifiable matter, following the methods given in the books on paints analysis. In some cases it may be desirable to let a portion of the well-mixed paint stand until the pigment settles out (or settle by means of a centrifuge), decant the vehicle, distil off the thinner, and examine the nonvolatile vehicle.

3. On an extracted and dried pigment, this determination is of little value. If the original paint contained gypsum, a part of the combined H_2O of the latter will be driven off in the drying of the extracted pigment and in the "moisture" determination.

4. This determination may serve as a rough or approximate check in many cases on the CO_2, H_2O, etc.

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[1] 1924 Book of A.S.T.M. Standards.

5. If the original sample contained $BaCO_3$ and $PbSO_4$, $CaSO_4$ or other soluble sulfate, the soluble Ba will form with the soluble sulfate a precipitate of $BaSO_4$ which will be determined as "insoluble matter." If the sample contained $SrSO_4$ or $SrCO_3$, some $SrSO_4$ may be counted as $BaSO_4$, some Sr will count as soluble barium, and some may be counted as CaO. This element is not separated, as it probably will not be encountered, or will be present as an impurity in the Ba and Ca compounds.

6. Any soluble Al_2O_3 (Fe_2O_3) and in most cases MgO, and sometimes some CaO, come from the siliceous pigment used. MgO generally denoted the presence of asbestine.

7. It is not possible to determine the amount of basic lead carbonate and lead sulfate when carbonates or soluble sulfates of other metals, such as calcium, are present. Also neither basic lead carbonate nor basic lead sulfate are definite compounds.

8. This will include any $BaSO_4$ that may have been dissolved as such. The weighed precipitate should be tested for $CaSO_4$, and if present, it should be removed by treating with hot dilute HCl, filtering, washing, igniting, and again weighing.

9. For very accurate work, or when the precipitate is large, the precipitate should be dissolved in HCl (1:1) and the precipitation repeated.

10. This precipitate may also contain Fe_2O_3, TiO_2, and P_2O_5.

11. If the sample contains antimony, it should be precipitated by H_2S in the hot acid solution, filtered off, washed, and the filtrate neutralized, etc., for zinc. The H_2S precipitate may also contain PbS. If no sulfide separation is made, any cadmium present will be counted as zinc.

12. Care must be exercised in this washing, as 1000 cc. of boiling water will dissolve over 0.01 g. of CaC_2O_4.

13. For more accurate work, the CaC_2O_4 precipitate should be ignited, cooled, cautiously moistened with water, redissolved in HCl and the solution diluted to 100 cc. Then NH_4OH should be added in slight excess, the liquid boiled, and filtered and washed if a precipitate appears. Then reprecipitate the Ca with NH_4OH and $(NH_4)_2C_2O_4$ as above, filter, wash, ignite, cool, and weigh; or, titrate as described.

14. The less the amount of magnesium present, the longer the precipitate must be allowed to settle.

15. If the sample contained manganese, it will be caught in large part with the $Mg_2P_2O_7$. If desired, Mn may be determined by dissolving the $Mg_2P_2O_7$ in HNO_3 and applying the bismuthate method.

16. If the sample is high in sulfide, *e. g.*, contains a high percentage of lithopone, grind 1 to 2 g. of the pigment with dry $K_2Cr_2O_7$, transfer to the evolution flask, add 50 cc. of water, and run in H_2SO_4 (1:1) from the separatory funnel. Or, place at the front of the purifying and drying train a tube containing acidified $CuSO_4$ solution, $KMnO_4$ solution, or CrO_3 solution.

17. The percentage of sulfide sulfur can be calculated from the percentages of total zinc and zinc soluble in 2 to 3 per cent acetic acid, assuming the sulfide to be ZnS. See Method (*d*) under determination of zinc.

18. This method is not applicable in the presence of sulfides decomposable under the conditions given.

19. A Knorr CO_2 apparatus is very convenient. In this case, the vertical condenser may be connected with an absorption tower containing the iodine solution, followed by the KI solution in a suitable tube.

20. To minimize, if not eliminate, any possible oxidation by the air, add about

1 g. (in one piece) of $NaHCO_3$ to the evolution flask, then add the acid directly to the flask, omitting the separatory funnel and the current of air. Boil the solution until about 50 cc. of distillate has passed over.

CALCULATIONS

The calculation of the component pigments of a mixed or combination pigment may be a somewhat difficult matter. Certain assumptions must be made, depending upon the complexity of the mixed pigment, as to the composition or formulas of component pigments and as to the manner in which the acidic and basic radicles are combined. Add any Al_2O_3 (Fe_2O_3) found in the soluble portion to the siliceous matter and report the sum as "Insoluble siliceous matter," unless the soluble Al is high; in this case, an aluminate is probably present, and the Al_2O_3 should be reported as Al_2O_3. If a small amount of soluble Mg is found, it should also be added to the siliceous matter. If the soluble Mg is high, the presence of $MgCO_3$ is indicated, and the MgO is calculated to $MgCO_3$ as pointed out below. The insoluble siliceous matter reported should be based on the weight obtained on drying the total insoluble matter at 105° C. if the combined H_2O contained therein is to be considered.

In the absence of ZnS or TiO_2, report $BaSO_4$ as $BaSO_4$. If ZnS is present, calculate the $BaSO_4$ equivalent by multiplying by 2.85; report sum of $ZnS+BaSO_4$ as "lithopone." If TiO_2 is present, calculate the $BaSO_4$ equivalent by multiplying by 3.17; report sum of TiO_2+BaSO_4 as "titanium pigment." Report residual $BaSO_4$ as $BaSO_4$. If TiO_2 is present and $BaSO_4$ is absent or is present in a smaller amount than would be indicated by the above factor, then report TiO_2 as TiO_2, and $BaSO_4$ as $BaSO_4$. If $CaCO_3$, $CaSO_4$, $BaCO_3$, and $MgCO_3$ are absent calculate CO_2 to basic carbonate white lead, $(PbCO_3)_2 \cdot Pb(OH)_2$, and soluble SO_3 to $PbSO_4$. Any excess of Pb is calculated to PbO, added to the $PbSO_4$, and the sum reported as basic lead sulfate; or, multiply the sum of $PbSO_4+PbO$ by 0.058 to obtain the ZnO; add this result to the $PbSO_4+PbO$ and report as basic sulfate white lead. (The ZnO factor is based on the assumption that the average composition of commercial basic sulfate white lead is: 78.5 per cent $PbSO_4$, 16.0 per cent PbO, and 5.5 per cent ZnO.) Lead oxide (PbO) should not be reported except in the presence of $PbSO_4$; unless the entire analysis is reported in the elementary or oxide form.

If the sample contains CO_2 but no soluble SO_3, calculate total Pb to basic carbonate white lead, $(PbCO_3)_2 \cdot Pb(OH)_2$; calculate residual CO_2 to $CaCO_3$, then to $BaCO_3$ and $MgCO_3$ if soluble Ba and Mg should be present in sufficient amounts to indicate the presence of these carbonates. The CO_2 result will be an index of this. A small amount of residual CaO is probably from the sliceous matter and should be added to the insoluble siliceous matter.

A small amount of soluble Ba may be from the $CaCO_3$ used or may be due to the solubility of $BaSO_4$ if this compound is present in the original pigment. This Ba may be calculated to $BaSO_4$ and added to the $BaSO_4$ found in the insoluble matter.

If the sample contains soluble SO_3 but no CO_2, calculate CaO to $CaSO_4$ or $CaSO_4 \cdot 2H_2O$; residual SO_3 to $PbSO_4$; add residual PbO to $PbSO_4$ and report sum as basic lead sulfate; or, multiply $PbSO_4 + PbO$ by 0.058 and add the result to the $PbSO_4 + PbO$, and report the total as basic sulfate white lead.

If the sample contains $CaCO_3$ ($MgCO_3$, $BaCO_3$) and also basic sulfate white lead, or $CaSO_4$ and basic carbonate white lead; or a mixture of these; it is not possible to determine or calculate the amount of $PbCO_3$ or $PbSO_4$ with any degree of certainty (Notes 3 and 5). The presence of appreciable amounts of CaO and SO_3 in the water-soluble matter indicates the probable presence of $CaSO_4$ in the original pigment. The following arbitrary calculations may be made; calculate water-soluble SO_3 to $CaSO_4$ or $CaSO_4 \cdot 2H_2O$, subtract this SO_3 from total soluble SO_3 and calculate the remainder to $PbSO_4$; calculate residual CaO to $CaCO_3$, and then residual CO_2 to $(PbCO_3)_2 \cdot Pb(OH)_2$. If there is an excess of CO_2, calculate to $MgCO_3$ or $BaCO_3$, if the amounts of soluble Mg and Ba indicate the probable presence of these carbonates. Add residual PbO to $PbSO_4$ and calculate, as above, to basic sulfate white lead. The procedure followed by the Federal Specifications Board should be noted.[1]

Report total antimony as Sb_2O_3.

Calculate sulfide sulfur to ZnS, subtract the Zn equivalent to the S from the total Zn, then subtract the Zn required for the basic sulfate white lead, and report the remainder as ZnO.

Report moisture, loss on ignition, So_2, and matter soluble in water directly.

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[1] Federal Specifications Board Specification No. 10 for "White Paint and Tinted Paints Made on a White Base, Semipaste and Ready Mixed"; Bureau of Standards *Circular No. 89*, 2d Ed., p. 2: "The total lead dissolved by dilute acetic acid and hot acid, ammonium acetate, weighed as lead sulfate, and this weight multiplied by the factor 0.883 shall be considered white lead. (It is not possible to determine the amount of lead carbonate and lead sulfate when carbonates or sulfates of other metals, such as calcium, are present. Also neither basic lead carbonate nor basic lead sulfate are definite compounds. The factor to convert $PbSO_4$ to $(PbCO_3)_2Pb(OH)_2$ is 0.854, to convert $PbSO_4$ to $PbSO_4 \cdot PbO$ is 0.868, and to convert $PbSO_4$ to $(PbSO_4)_2 \cdot PbO$ is 0.913. The arbitrary factor used under this specification is the mean of the largest and smallest of these three factors.)"

TENTATIVE METHOD OF TEST
FOR
DISTILLATION OF GASOLINE, NAPHTHA, KEROSINE, AND SIMILAR PETROLEUM PRODUCTS[1]

Serial Designation: D 86 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921; Revised, 1923, 1924, 1925.

APPARATUS

Flask.

1. *Flask.*—The Standard 100-cc. Engler flask is shown in Fig. 1, the dimensions and allowable tolerance being as follows:

	Centimeters	Inches	Tolerances, cm.
Diameter of bulb, outside	6.5	2.56	0.2
" " neck, inside	1.6	0.63	0.1
Length of neck	15.0	5.91	0.4
" " vapor tube	10.0	3.94	0.3
Diameter of vapor tube, outside	0.6	0.24	0.05
" " " " inside	0.4	0.16	0.05
Thickness of vapor tube wall	0.1	0.04	0.05

The position of the vapor tube shall be 9 cm. (3.55 in.) ±3mm. above the surface of the liquid when the flask contains its charge of 100 cc. The tube is approximately in the middle of the neck and set at an angle of 75 deg. (tolerance ±3 deg.) with the vertical.

Condenser.

2. *Condenser.*—The condenser (Fig. 2) consists of a $\frac{9}{16}$-in. (14.29 mm.) OD No. 20 Stubbs Gage seamless brass tube, 22 in. (55.88 cm.) long. It is set at an angle of 75 deg. from the perpendicular and is surrounded with a cooling bath 15 in. long (38.1 cm.), approximately 4 in. (10.16 cm.) wide by 6 in. (15.24 cm.) high. The lower end of the condenser tube is cut off at an acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the graduate at a point 1 to $1\frac{1}{4}$ in. (2.54 to 3.8 cm.) below the top of the graduate when it is in position to receive the distillate.

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

Shield.

3. *Shield.*—The shield (Fig. 2) is made of approximately 22 gage sheet metal and is 19 in. (48.26 cm.) high, 11 in. (27.94 cm.) long and 8 in. (20.32 cm.) wide, with a door on one narrow side, with two openings, 1 in. (2.54 cm.) in diameter, equally spaced, in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings are 8½ in. (21.59 cm.) below the top of the shield. There are also three ½-in. (1.27 cm.) holes in each of the four sides with their centers 1 in. (2.54 cm.) above the base of the shield.

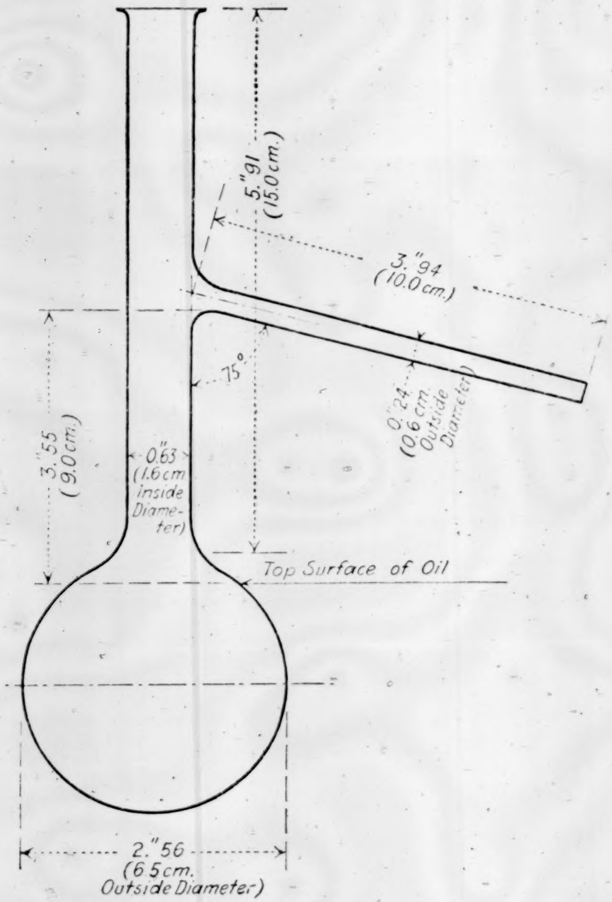
5."91 (15.0 cm.)
3."94 (10.0 cm.)
75°
0."24 (0.6 cm. Outside Diameter)
3."55 (9.0 cm.)
0."63 (1.6 cm inside Diameter)
Top Surface of Oil
2."56 (6.5 cm. Outside Diameter)

FIG. 1.

Support.

4. *Ring Support and Hard Asbestos Boards.*—The ring support is of the ordinary laboratory type, 4 in. (10.16 cm.) or larger in diameter, and is supported on a stand inside the shield. There are two hard asbestos boards: One 6 by 6 by $\frac{1}{4}$ in. (15.24 by 15.24 cm. by 6.35 mm.) with a hole $1\frac{1}{4}$ in. (3.175 cm.)[1] in diameter in its center, the sides of which shall be perpendicular to the surface; the other, an asbestos board to fit tightly inside the shield, with an opening 4 in. (10.16 cm.) in diameter concentric with the ring support. These are arranged as follows: The second asbestos board is placed on the ring and the first or smaller asbestos board on top so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat is applied to the flask only through the $1\frac{1}{4}$-in. (3.175 cm.) opening in the first asbestos board.

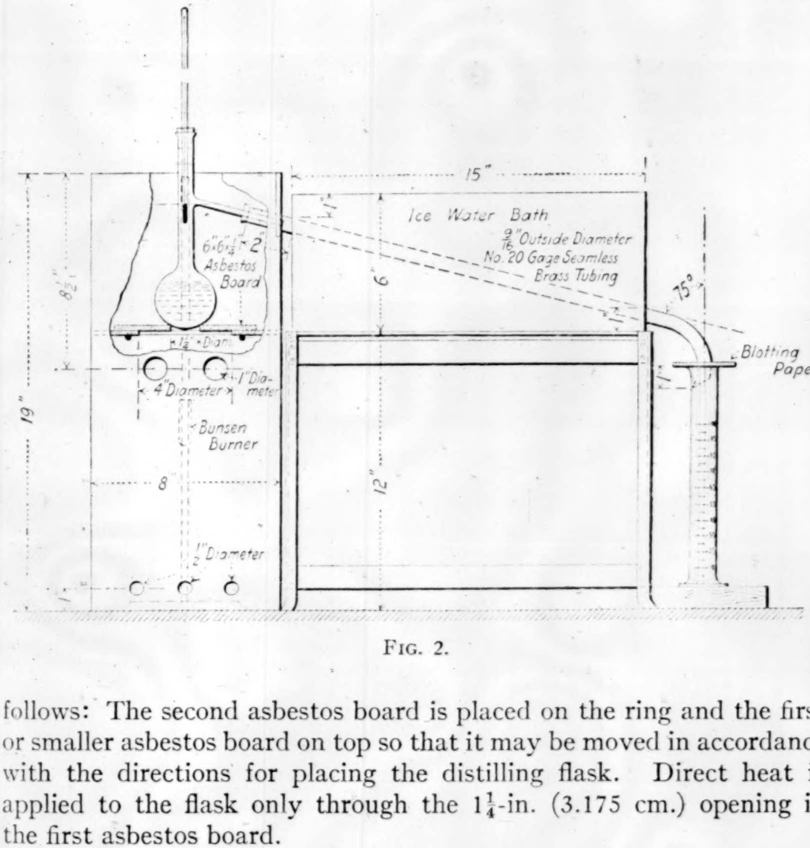
15"
Ice Water Bath
9/16" Outside Diameter
No. 20 Gage Seamless
Brass Tubing
1"
6"
75°
Blotting Paper
6"×6"×1/4"
2"
Asbestos
Board
8 1/2"
19"
1 1/4" Diam.
4" Diameter
1" Diameter
Bunsen
Burner
8"
12"
1/2" Diameter
1"

FIG. 2.

Heater.

5. *Gas Burner or Electric Heater.*—

(*a*) *Gas Burner.*—The burner is so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified below. The flame should never be so large that it spreads over a circle of diameter greater than 3½ in. (8.89 cm.) on the under surface of the asbestos board. A sensitive regulating valve is a necessary adjunct, as it gives complete control of heating.

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[1] When distilling petroleum products having an end point above 470° F. (243.34° C.), the hole in the asbestos board shall be $1\frac{1}{2}$ in. (3.81 cm.) in diameter.

(*b*) *Electric Heater.*—The electric heater, which may be used in place of the gas flame, shall be capable of bringing over the first drop within the time specified below when started cold, and of continuing the distillation at the uniform rate. The electric heater shall be fitted with an asbestos board top ⅛ to ¼ in. (3.175 to 6.35 mm.) thick, having a hole 1¼ in. (3.175 cm.) in diameter in the center. When an electric heater is employed, the portion of the shield above the asbestos board shall be the same as with the gas burner but the part below the board may be omitted.

Thermometers.

6. *Thermometers.*—

(*a*) The A.S.T.M. Low-Distillation Thermometer shall conform to the following requirements. These specifications cover a total-immersion thermometer graduated in either Centigrade or Fahrenheit degrees, as specified, the ranges being 0 to 300° C. or 30 to 580° F., respectively.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISION: 0 to 300° C. in 1° C. or 30 to 580° F. in 2° F.

TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).

STEM: Plain front, enamel back, suitable thermometer tubing.
Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 10 to 15 mm. (0.39 to 0.59 in.).
Diameter, 5.0 to 6.0 mm. (0.20 to 0.24 in.).

DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 100 to 110 mm. (3.94 to 4.33 in.).

DISTANCE TO 300° C. OR 572° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

IMMERSION: Total.

SPECIAL MARKING: "A.S.T.M. Low Distillation," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.5° C. or 1° F.

STANDARDIZATION: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 300° C. or 572° F.

TEST FOR PERMANENCY OF RANGE: After being subjected to a temperature of 280 to 290° C. or 540 to 560° F. for 24 hours, the accuracy shall be within the limit specified.

Case: The thermometer shall be supplied in a suitable case on which shall appear The marking: "A.S.T.M. Low Distillation, 0 to 300° C." or "A.S.T.M. Low Distillation, 30 to 580° F." according to the type of thermometer.

Note.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

(*b*) The A.S.T.M. High-Distillation Thermometer shall conform to the following requirements. These specifications cover a total-immersion thermometer graduated either in Centigrade or Fahrenheit degrees, as specified, the ranges being 0 to 400° C. or 30 to 760° F., respectively.

Type: Etched stem, glass.

Liquid: Mercury.

Range and Subdivision: 0 to 400° C. in 1° C. or 30 to 760° F. in 2° F.

Total Length: 378 to 384 mm. (14.88 to 15.12 in.).

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

Bulb: Corning normal or equally suitable thermometric glass.
Length, 10 to 15 mm. (0.39 to 0.59 in.).
Diameter, 5.0 to 6.0 mm. (0.20 to 0.24 in.).

Distance to 0° C. or 32° F. Line from Bottom of Bulb: 25 to 35 mm. (0.98 to 1.38 in.).

Distance to 400° C. or 752° F. Line from Top of Thermometer: 30 to 45 mm. (1.18 to 1.77 in.).

Filling Above Mercury: Nitrogen gas.

Top Finish: Glass ring.

Graduation: All lines, figures and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

Immersion: Total.

Special Marking: "A.S.T.M. High Distillation" a serial number and the manufacturer's name or trade mark shall be etched on the stem.

Scale Error: The error at any point of the scale up to 370° C. or 700° F. when the thermometer is standardized as provided below shall not exceed 1° C. or 2° F.

Standardization: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 370° C. or 700° F.

Test for Permanency of Range: After being subjected to a temperature between 360 and 370° C. or 680 and 700° F. for 24 hours, the accuracy shall be within the limit specified.

Case: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. High Distillation, 0 to 400° C." or "A.S.T.M. High Distillation, 30 to 760° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

Graduate. 7. *Graduate.*—The graduate shall be of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 cc., and the graduated portion shall be not less than 7 in. (17.78 cm.) nor more than 8 in. (20.32 cm.) long. It shall be graduated in single cubic centimeters and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up at intervals of 10 cc. The overall height of the graduate shall be not less than 9¾ in. (24.8 cm.), nor more than 10¼ in. (26.0 cm.). The graduations shall not be in error by more than 1 cc. at any point on the scale.

PROCEDURE

Procedure. 8. (*a*) The condenser bath shall be filled with cracked ice,[1] and enough water added to cover the condenser tube. The temperature shall be maintained between 32 and 40° F. (0 and 4.45° C.).

(*b*) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft cloth attached to a cord or copper wire may be used for this purpose.

(*c*) One hundred cubic centimeters of the product shall be measured in the 100-cc. graduated cylinder at 55 to 65° F. (12.78 to 18.33° C.) and transferred directly to the Engler flask. None of the liquid shall be permitted to flow into the vapor tube.

(*d*) The thermometer[2] provided with a cork shall be fitted tightly into the flask so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask.

(*e*) The charged flask shall be placed in the 1¼-in. (3.175-cm.) opening in the 6 by 6-in. (15.24 by 15.24-cm.) asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the vapor tube extends into the condenser tube not less than 1 in. (2.54 cm.) nor more than 2 in. (5.08 cm.).

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[1] Any other convenient cooling medium may be used.

[2] For products having an initial boiling point of 212° F. (100° C.) or higher, the High-Distillation Thermometer shall be used; for all other products, the Low-Distillation Thermometer shall be used.

(*f*) The graduated cylinder used in measuring the charge shall be placed, without drying, at the outlet of the condenser tube in such a position that the condenser tube shall extend into the graduate at least 1 in. (2.54 cm.) but not below the 100-cc. mark. Unless the temperature is between 55 and 65° F. (12.78 and 18.33° C.) the receiving graduate shall be immersed up to the 100-cc. mark in a transparent bath maintained between these temperatures. The top of the graduate shall be covered closely during the distillation with a piece of blotting paper or its equivalent, cut so as to fit the condenser tube tightly.

Distillation.

9. When everything is in readiness, heat shall be applied at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 5 nor more than 10 minutes. When the first drop falls from the end of the condenser the reading of the distillation thermometer shall be recorded as the *initial boiling point.* The receiving cylinder shall then be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the distillation will proceed at a uniform rate of not less than 4 nor more than 5 cc. per minute. The volume of distillate collected in the cylinder shall be observed and recorded, to the nearest 0.5 cc., when the mercury of the thermometer reaches each point that is an even multiple of 25° C. or the Fahrenheit equivalent of this point (50° C., 75° C., 100° C., 125° C., etc., or 122° F., 167° F., 212° F., 257° F., etc.).

When the liquid residue in the distillation flask is approximately 5 cc. the heat may be increased because of the presence of heavy ends which have relatively high boiling points. However, no further increase of heat shall be applied after this adjustment. The 4 to 5-cc. rate can rarely be maintained from this point to the end of the distillation, but in no case shall the period between the point when approximately 5 cc. of liquid remains in the flask and the end point be more than 5 minutes.

The heating shall be continued until the mercury reaches a maximum and starts to fall consistently. The highest temperature observed on the distillation thermometer shall be recorded as the *maximum temperature* or end point. Usually this point will be reached after the bottom of the flask has become dry.

The total volume of the distillate collected in the receiving graduate shall be recorded as the *recovery.*

The cooled residue shall be poured from the flask into a small cylinder graduated in 0.1 cc., measured when cool and the volume recorded as *residue.*

The difference between 100 cc. and the sum of the recovery and the residue shall be calculated and recorded as *distillation loss*.

ACCURACY

Accuracy.

10. With proper care and attention to detail, duplicate results obtained for initial boiling point and maximum temperature, respectively, should not differ from each other by more than 6° F. (3.33° C.).

CORRECTION FOR BAROMETRIC PRESSURE

11. The actual barometric pressure shall be ascertained and recorded, but no correction shall be made except in case of dispute. In such cases the temperature points shall be corrected to 760 mm. (29.92 in.), by the use of the Sydney Young equation, as follows:

For centigrade readings:

$$C_c = 0.00012\,(760-P)\,(273+t_c)$$

For Fahrenheit readings:

$$C_f = 0.00012\,(760-P)\,(460+t_f)$$

in which C_c and C_f are, respectively, corrections to be added to the observed temperature t_c or t_f, and P is the actual barometric pressure in millimeters of mercury.

The following table is a convenient approximation of the corrections as calculated by the above equation.

Temperature Range		Correction[1] per 10-mm. Difference in Pressure,	
deg. Cent.	deg. Fahr.	deg. Cent.	deg. Fahr.
10 - 30	50 - 86	0.35	0.63
30 - 50	86 - 122	0.38	0.68
50 - 70	122 - 158	0.40	0.72
70 - 90	158 - 194	0.42	0.76
90 - 110	194 - 230	0.45	0.81
110 - 130	230 - 266	0.47	0.85
130 - 150	266 - 302	0.50	0.89
150 - 170	302 - 338	0.52	0.94
170 - 190	338 - 374	0.54	0.98
190 - 210	374 - 410	0.57	1.02
210 - 230	410 - 446	0.59	1.06
230 - 250	446 - 482	0.62	1.11
250 - 270	482 - 518	0.64	1.15
270 - 290	518 - 554	0.66	1.19
290 - 310	554 - 590	0.69	1.24
310 - 330	590 - 626	0.71	1.28
330 - 350	626 - 662	0.74	1.32
350 - 370	662 - 698	0.76	1.37
370 - 390	698 - 734	0.78	1.41
390 - 410	734 - 770	0.81	1.45

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[1] To be added in case barometric pressure is below 760 mm.; to be subtracted in case barometric pressure is above 760 mm.

TENTATIVE METHOD OF TEST
FOR
DISTILLATION OF NATURAL GAS GASOLINE[1]

Serial Designation: D 216 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. **It is not a Standard of the Society and is subject to annual revision.**

ISSUED, 1925

I. APPARATUS

Flask. 1. *Flask.*—The standard 100-cc. Engler flask is shown in Fig. 1, the dimensions and allowable tolerance being as follows:

	CENTIMETERS	INCHES	TOLERANCES, CM.
Diameter of bulb, outside	6.5	2.56	0.2
Diameter of neck, inside	1.6	0.63	0.1
Length of neck	15.0	5.91	0.4
Length of vapor tube	10.0	3.94	0.3
Diameter of vapor tube, outside	0.6	0.24	0.05
Diameter of vapor tube, inside	0.4	0.16	0.05
Thickness of vapor tube wall	0.1	0.04	0.05

The position of the vapor tube shall be 9 cm. (3.55 in.) ± 3 mm. above the surface of the liquid when the flask contains its charge of 100 cc. The tube is approximately in the middle of the neck and set at an angle of 75 deg. (tolerance ± 3 deg.) with the vertical.

Condenser. 2. *Condenser.*—The condenser (Fig. 2) consists of a $\frac{9}{16}$-in. (14.29-mm.) OD No. 20 Stubbs Gage seamless brass tube, 22 in. (55.88 cm.) long. It is set at an angle of 75 deg. from the perpendicular and is surrounded with a cooling bath 15 in. long (38.1 cm.) approximately 4 in. (10.16 cm.) wide by 6 in. (15.24 cm.) high. The lower end of the condenser tube is cut off at an acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the graduate at a point 1 to $1\frac{1}{4}$ in. (2.54 to 3.8 cm.) below the top of the graduate when it is in position to receive the distillate.

Shield. 3. *Shield.*—The shield (Fig. 2) is made of approximately No. 22 gage sheet metal and is 19 in. (48.26 cm.) high, 11 in. (27.94 cm.) long and 8 in. (20.32 cm.) wide, with a door on one narrow side, with two openings, 1 in. (2.54 cm.) in diameter equally spaced, in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings are 8½ in. (21.59 cm.) below the top of the shield. There are also three ½ in. (1.27 cm.) holes in each of the four sides with their centers 1 in. (2.54 cm.) above the base of the shield.

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

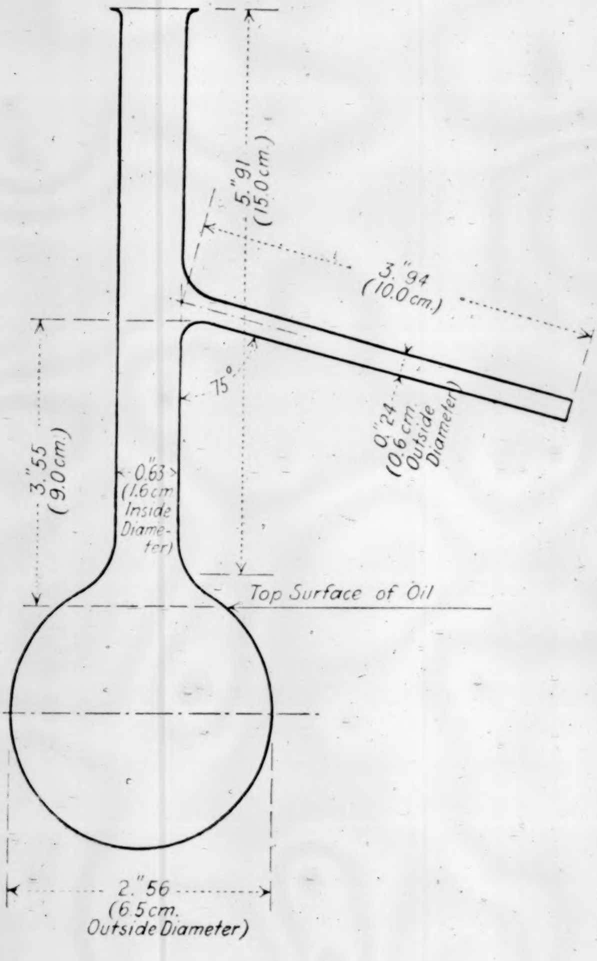
5."91 (15.0 cm.)
3."94 (10.0 cm.)
75°
0."24 (0.6 cm. Outside Diameter)
3."55 (9.0 cm.)
0."63 (1.6 cm Inside Diameter)
Top Surface of Oil
2."56 (6.5 cm. Outside Diameter)

FIG. 1.

Support. 4. *Ring Support and Hard Asbestos Boards.*—The ring support is of the ordinary laboratory type, 4 in. (10.16 cm.) or larger in diameter, and is supported on a stand inside the shield. There are two hard asbestos boards: one 6 by 6 by $\frac{1}{4}$ in. (15.24 by 15.24 cm. by 6.35 mm.), with a hole $1\frac{1}{4}$ in. (3.175 cm.) in diameter in its center, the sides of which shall be perpendicular to the surface; the other, to fit tightly inside the shield, with an opening 4 in. (10.16 cm.) in diameter concentric with the ring support. These are arranged as follows: The second asbestos board is placed on the ring, and the first or smaller asbestos board on top so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat is applied to the flask only through the $1\frac{1}{4}$ in. (3.175 cm.) opening in the first asbestos board.

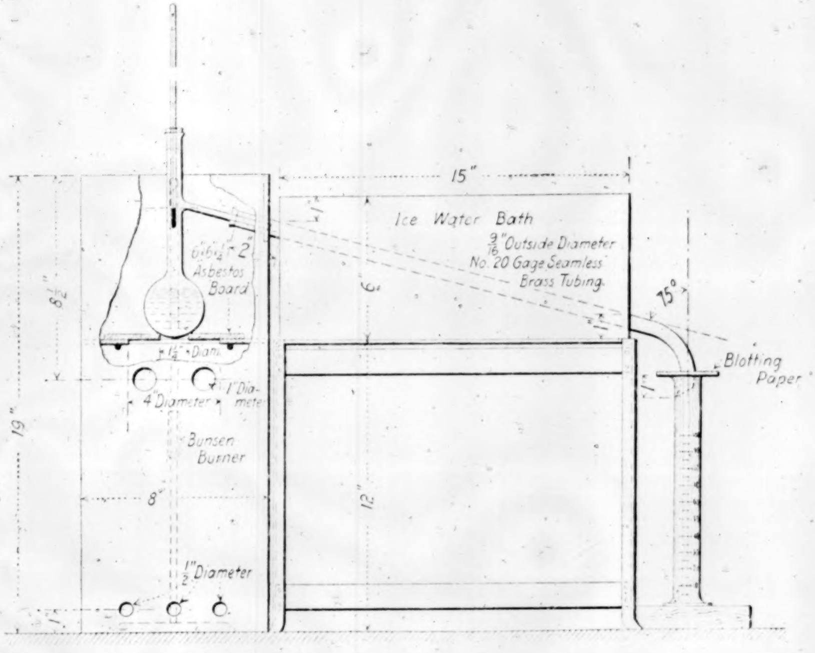
15"
19"
8½"
6"×6"×¼"–2" Asbestos Board
1¼" Diam.
4" Diameter
1" Diameter
Bunsen Burner
8"
½" Diameter
1"
Ice Water Bath
9/16" Outside Diameter No. 20 Gage Seamless Brass Tubing.
1"
6"
12"
75°
1"
1"
Blotting Paper

FIG. 2.

5. *Gas Burner or Electric Heater.*

Heater.

(*a*) *Gas Burner.*—The burner is so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified below. The flame should never be so large that it spreads over a circle of a diameter greater than $3\frac{1}{2}$ in. (8.89 cm.) on the under surface of the asbestos board. A sensitive regulating valve is a necessary adjunct, as it gives complete control of heating.

(*b*) *Electric Heater.*—The electric heater, which may be used in place of the gas flame, shall be capable of bringing over the first drop within the time specified below when started cold, and of continuing the distillation at the uniform rate. The electric heater shall be fitted with an asbestos board top $\frac{1}{8}$ to $\frac{1}{4}$ in. (3.175 to 6.35 mm.) thick, having a hole $1\frac{1}{4}$ in. (3.175 cm.) in diameter in the center. When an electric heater is employed, the portion of the shield above the asbestos board shall be the same as with the gas burner but the part below the board may be omitted.

Thermometer.

6. *Thermometer.*—The A.S.T.M. Low-Distillation Thermometer shall conform to the following requirements. These specifications cover a total-immersion thermometer graduated in either Centigrade or Fahrenheit degrees, as specified, the ranges being 0 to 300° C. or 30 to 580° F., respectively.

Type: Etched stem, glass.

Liquid: Mercury.

Range and Subdivision: 0 to 300° C. in 1° C. or 30 to 580° F. in 2° F.

Total Length: 378 to 384 mm. (14.88 to 15.12 in.).

Stem: Plain front, enamel back, suitable thermometer tubing.
Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

Bulb: Corning normal or equally suitable thermometric glass.
Length, 10 to 15 mm. (0.39 to 0.59 in.).
Diameter, 5.0 to 6.0 mm. (0.20 to 0.24 in.).

Distance to 0° C. or 32° F. Line from Bottom of Bulb: 100 to 110 mm. (3.94 to 4.33 in.).

Distance to 300° C. or 572° F. Line from Top of Thermometer: 30 to 45 mm. (1.18 to 1.77 in.).

Filling above Mercury: Nitrogen gas.

Top Finish: Glass ring.

Graduation: All lines, figures, and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

Immersion: Total.

Special Marking: "A.S.T.M. Low Distillation," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

Scale Error: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.5° C. or 1° F.

Standardization: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 300° C. or 572° F.

Test for Permanency of Range: After being subjected to a temperature of 280 to 290° C. or 540 to 560° F. for 24 hours, the accuracy shall be within the limit specified.

Case: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. Low Distillation, 0 to 300° C." or "A.S.T.M. Low Distillation, 30 to 580° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the overall length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

Graduate.

7. *Graduate.*—The graduate shall be of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 cc., and the graduated portion shall be not less than 7 in. (17.78 cm.) nor more than 8 in. (20.32 cm.) long. It shall be graduated in single cubic centimeters and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up, at intervals of 10 cc. The overall height of the graduate shall not be less than 9¾ in. (24.8 cm.), nor more than 10¼ in. (26.0 cm.). The graduations shall not be in error by more than 1 cc. at any point on the scale.

II. PROCEDURE

Sampling.

8. *Sampling.*—Samples should be collected in a previously cooled bottle, preferably by immersing the bottle in the liquid, where possible, and discarding the first sample. Where immersion is not possible the sample should be drawn off into a previously cooled bottle in such a manner that agitation is kept at a minimum. Immediately close the bottle with a tight-fitting stopper, and place in an ice bath or refrigerator capable of bringing the gasoline to a temperature of not less than 32° F. (0° C.), nor more than 40° F. (4.45° C.).

Procedure.

9. (*a*) The condenser bath shall be filled with cracked ice[1] and enough water added to cover the condenser tube. The temperature shall be maintained between 32 and 34° F. (0 and 1.1° C.). Agitation with air greatly aids in maintaining the required temperature range.

(*b*) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft cloth attached to a cord or copper wire may be used for this purpose.

(*c*) One hundred cubic centimeters of the product shall be measured in the 100-cc. graduated cylinder at 32 to 40° F. (0 to 4.45° C.) and transferred directly to the Engler flask. Both the flask and the graduated cylinder shall have been cooled to a temperature of from 32 to 40° F. (0 to 4.45° C.) before use. None of the liquid shall be permitted to flow into the vapor tube.

(*d*) The thermometer provided with a cork shall be fitted tightly into the flask so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask.

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[1] Any other convenient cooling medium may be used.

(*e*) The charged flask shall be placed in the 1¼-in. (3.175-cm.) opening in the 6 by 6-in. (15.24 by 15.24-cm.) asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the vapor tube extends into the condenser tube not less than 1 in. (2.54 cm.) nor more than 2 in. (5.08 cm.).

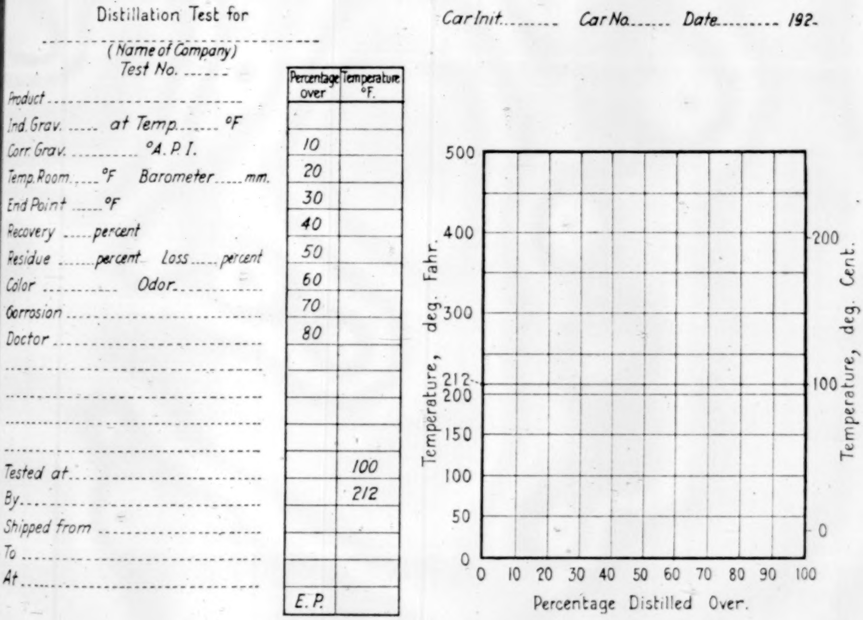
(*f*) The graduated receiver used in measuring the charge shall be placed, without drying, at the outlet of the condenser tube in such a position that the condenser tube shall extend into the graduate at least 1 in. (2.54 cm.), but not below the 100-cc. mark. The graduate shall be immersed at least up to the 100-cc. mark in a transparent bath maintained between the temperatures of 32 and 34° F. (0 and 1.11° C.) The top of the graduate shall be covered closely during the distillation with a piece of blotting paper, or its equivalent, and cut to fit the condenser tube tightly. A lead washer, or other suitable material, resting on the blotting paper is a convenient accessory as it serves to hold the paper tightly against the top of the graduate and also provides the additional weight necessary to overcome the buoyant effect of the liquid in the cooling bath.

10. When everything is in readiness, heat shall be applied at a uniform rate, so regulated that the first drop of the condensate falls from the condenser in not less than two nor more than five minutes. When the first drop falls from the end of the condenser, the receiving cylinder shall be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the first 10 cc. of distillate shall be recovered in not less than three nor more than four minutes. Thereafter, the rate of distillation shall be maintained uniformly at not less than four nor more than 5 cc. per minute. The reading of the distillation thermometer shall be recorded when the level of the distillate reaches each 10-cc. mark on the graduate.

When the liquid residue in the distillation flask is approximately 5 cc., the heat may be increased because of the presence of heavy ends which have relatively high boiling points. However, no further increase of heat shall be applied after this adjustment. The 4 to 5-cc. rate cannot always be maintained from this point to the end of the distillation, but in no case should the period between the point when approximately 5 cc. of the liquid remains in the flask and the end point be more than 5 minutes.

The heating shall be continued until the mercury reaches a maximum and starts to fall consistently. The highest temperature observed on the distillation thermometer shall be recorded as the end point. Usually this point will be reached after the bottom of the flask has become dry. If the bottom of the flask is not dry, the operator should note this on his report.

The total volume of the distillate collected in the receiving graduate shall be recorded as the "recovery."



Distillation Test for
....
(Name of Company)
Test No. ......
Product ....
Ind. Grav. ...... at Temp. .... °F
Corr. Grav. .... °A. P. I.
Temp. Room......°F Barometer.....mm.
End Point ......°F
Recovery .....percent
Residue ....percent Loss.....percent
Color .... Odor..........
Corrosion ....
Doctor ....
....
....
....
....
Tested at.........................
By.........................
Shipped from ....
To ....
At ....

Car Init......... Car No....... Date......... 192-

Percentage over	Temperature °F.
10	
20	
30	
40	
50	
60	
70	
80	
	100
	212
E. P.	

500
400
300
212
200
150
100
50
0
Temperature, deg. Fahr.
200
100
0
Temperature, deg. Cent.
0 10 20 30 40 50 60 70 80 90 100
Percentage Distilled Over.

FIG. 3.—Form for Recording Tests on Natural Gas Gasoline.

The cooled residue shall be poured from the flask into a small cylinder graduated in 0.1 cc., measured when cool, and the volume recorded as "residue" at 32 to 40° F. (0 to 4.45° C.).

The difference between 100 cc. and the sum of the recovery and the residue shall be calculated and recorded as "distillation loss."

Correction.

11. *Correction for Barometric Pressure.*—The actual barometric pressure shall be ascertained and recorded, but no correction shall be made except in case of dispute. In such cases the temperature points shall be corrected to 760 mm. (29.92 in.), by the use of the Sydney Young equation, as follows:

For Centigrade readings:

$$C_c=0.00012\ (760-P)\ (273+t_c),$$

For Fahrenheit readings:

$$C_f=0.00012\ (760-P)\ (460+t_f),$$

in which C_c and C_f are, respectively, corrections to be added to the observed temperature t_c or t_f, and P is the actual barometric pressure in millimeters of mercury.

The following table is a convenient approximation of the corrections as calculated by the above equation.

TEMPERATURE RANGE		CORRECTION[1] PER 10-MM. DIFFERENCE IN PRESSURE,	
DEG. CENT.	DEG. FAHR.	DEG. CENT	DEG. FAHR.
10 – 30	50 – 86	0.35	0.63
30 – 50	86 – 122	0.38	0.68
50 – 70	122 – 158	0.40	0.72
70 – 90	158 – 194	0.42	0.76
90 – 110	194 – 230	0.45	0.81
110 – 130	230 – 266	0.47	0.85
130 – 150	266 – 302	0.50	0.89
150 – 170	302 – 338	0.52	0.94
170 – 190	338 – 374	0.54	0.98
190 – 210	374 – 410	0.57	1.02
210 – 230	410 – 446	0.59	1.06
230 – 250	446 – 482	0.62	1.11
250 – 270	482 – 518	0.64	1.15
270 – 290	518 – 554	0.66	1.19
290 – 310	554 – 590	0.69	1.24
310 – 330	590 – 626	0.71	1.28
330 – 350	626 – 662	0.74	1.32
350 – 370	662 – 698	0.76	1.37
370 – 390	698 – 734	0.78	1.41
390 – 410	734 – 770	0.81	1.45

Recording Results. 12. The form shown in Fig. 3 is suggested for the recording of results of tests of natural gas gasoline.

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[1] To be added in case barometric pressure is below 760 mm., to be subtracted in case barometric pressure is above 760 mm.

TENTATIVE METHOD OF TEST
FOR
PENETRATION OF GREASES[1]

Serial Designation: D 217 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Interpretation of Results.

It should be understood that the original consistency of grease is determined by a variety of factors which are very difficult to control. The soap content is the most important single factor, but the kind of fats used, the method of mixing, the final water content, and the temperature of pouring are all very important in determining the final consistency, so that it is impracticable to maintain these consistencies within narrow limits. Any working of the sample after it is poured, or any remelting, will greatly change the consistency. The original consistency also changes to some extent on standing, changing most rapidly during the first 48 hours after manufacture and more slowly thereafter.

Although most tests in the past have been based on the original consistency of greases, this consistency bears no necessary relationship to the worked consistency which is the factor of practical importance for most uses of cup greases, etc. Tests on such greases should therefore be made on the worked consistency. It is also almost impossible to measure the unworked consistency when samples have to be taken from barrels or other large containers.

On the other hand, for very hard greases, such as railroad greases used in slab form, the original or unworked consistency is generally the property of practical importance and should be the object of the test.

Scope.

1. This method shall be used to measure the original consistency or the worked consistency of No. 0 cup grease and all harder greases.

2. The method should be applied only to greases which have not been melted after having been poured and cooled during manufacture.

I. APPARATUS

Penetrometer.

3. The penetration shall be measured on what is customarily known as an asphalt penetrometer such as that described in the Proceedings of the American Society for Testing Materials, Vol. VII, p. 626 (1907), and illustrated in Fig. 3 herein. It is desirable, however, to modify this penetrometer by placing a flat metal plate or

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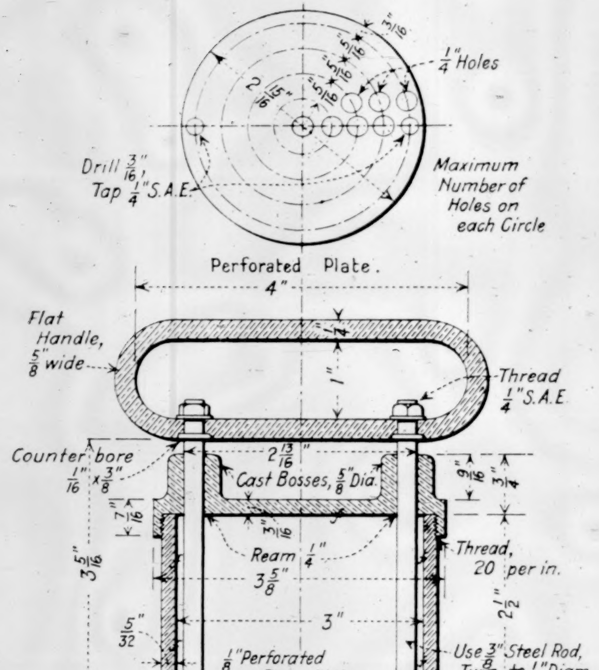
[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.



3/16"
5/16"
5/16"
5/16"
2 5/16"
1/4" Holes
Drill 3/16", Tap 1/4" S.A.E.
Maximum Number of Holes on each Circle
Perforated Plate.
4"
Flat Handle, 5/8" wide
1/4"
1"
Thread 1/4" S.A.E.
Counter bore 1/16" x 3/8"
2 13/16"
Cast Bosses, 5/8" Dia.
9/16"
3/4"
7/16"
3/16"
Ream 1/4"
Thread, 20 per in.
3 5/16"
3 5/8"
2 1/2"
5/32"
3"
1/8" Perforated Brass Plate
Use 3/8" Steel Rod, Turn to 1/4" Diam.
5/16"
4 1/4"
Thread 1/4" S.A.E.
Three Bolt Holes, Equal Spacing
Drill 7/16" and 5/16"; 1/2" C.L. to C.L.
4 1/4"
1 7/8" Rad.
3 5/16"
3"
All Fillets 1/8" Diameter
Material: Brass, except Rods.
Plan of Base.

FIG. 1.—Grease Worker.

transite board on top of the present grid base to give a flat support for the grease can. If many tests are to be made, it is preferable to replace the ordinary grid base with a special flat base 6 in. in diameter.

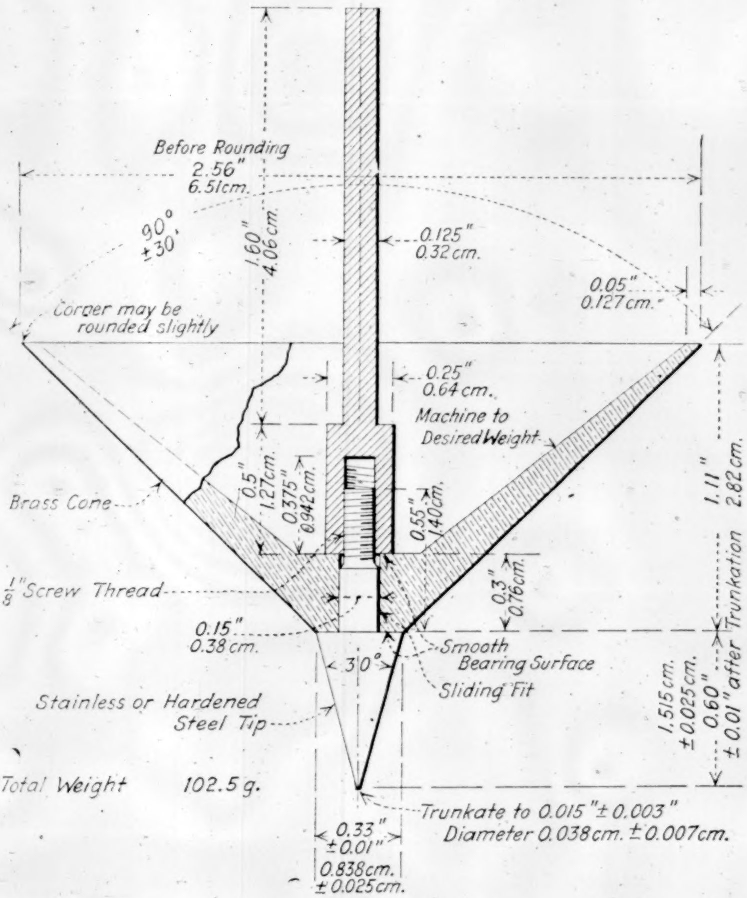

Before Rounding
2.56"
6.51cm.
90°
±30'
1.60"
4.06cm.
0.125"
0.32cm.
0.05"
0.127cm.
Corner may be rounded slightly
0.25"
0.64cm.
Machine to Desired Weight
Brass Cone
0.5"
1.27cm.
0.375"
0.942cm.
0.55"
1.40cm.
1.11"
2.82cm.
1/8" Screw Thread
0.3"
0.76cm.
0.15"
0.38cm.
30°
Smooth Bearing Surface
Sliding Fit
Stainless or Hardened Steel Tip
1.515cm.
±0.025cm.
0.60"
±0.01" after Trunkation
Total Weight 102.5 g.
Trunkate to 0.015" ± 0.003"
Diameter 0.038cm. ± 0.007cm.
0.33"
±0.01"
0.838cm.
±0.025cm.

FIG. 2.—Penetrometer Cone.

This plate should have a cork insert in the center to prevent dulling the tip of the cone if it should drop.

Cone and Plunger.

4. The needle used in the asphalt penetration tests shall be replaced with the cone shown in Fig. 2.[1] The cone shall be constructed of stainless steel or of brass with a detachable hardened steel or

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[1] The cone may be combined with the asphalt plunger, if desired, providing the total weight of cone and plunger and the outside dimensions of the cone are not thereby changed.

stainless steel tip and made in accordance with the outside dimensions and tolerances shown. The interior construction and dimensions without tolerances are intended to be suggestive only. The outside surface of the cone and tip shall be given a very smooth finish.

Weight. 5. The total moving weight in the test shall be 150 g. If the asphalt plunger weighs 47.5 g. (50 g. − 2.5 g. needle) the cone or the cone plus an attached weight must weigh 102.5 g. It is in general desirable to make the cone as light as possible so that it will have a wide range of usefulness, and to add a weight to give the desired total for the grease test. If the conditions of the tests are not specified, the penetration of a grease shall be understood to refer to the results of this test made at 77° F. (25° C.).

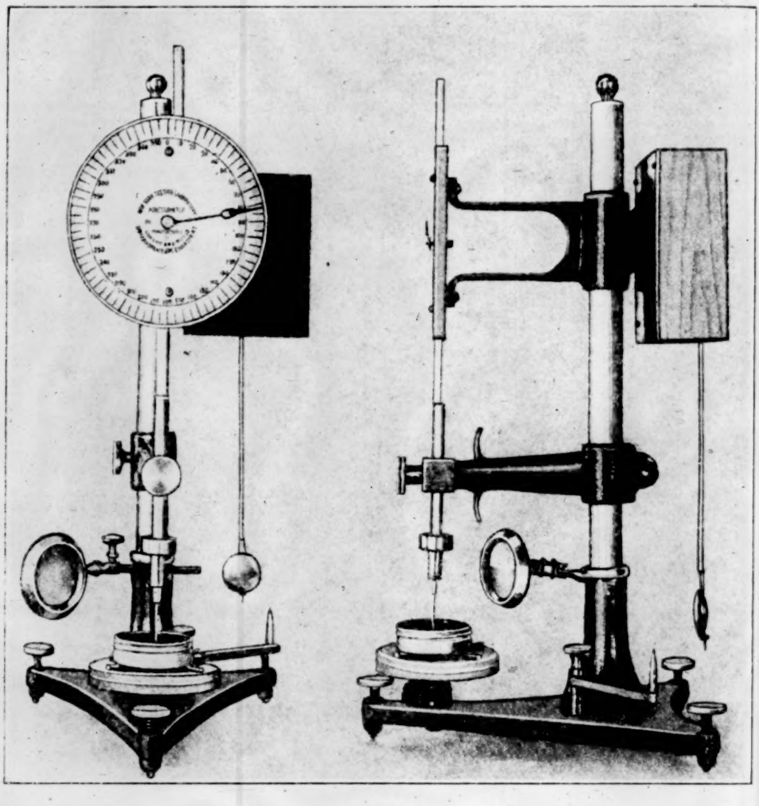

FIG. 3.—Penetrometer.

Grease-Worker. 6. The grease worker shown in Fig. 1 shall be used to work the grease to constant consistency in tests where the worked consistency is to be measured.

Constant Temperature Bath.

7. A constant temperature bath regulated to 77° F. ±1° F. (25° C. ±0.5° C.) is desirable to bring the samples to the temperature of test if many tests are to be made.

II. SAMPLES

8. Tests on the unworked consistency of grease shall be made only on grease in the original container (or cake in the case of very hard greases), 1-lb. tins being especially convenient for the purpose. If samples are to be taken from large containers, the tests must be made only on a sample worked as specified below. In taking samples from large containers, any discolored layers near the surface shall first be completely cut away and rejected.

III. PROCEDURE

(*A*) *Original* (*Unworked*) *Consistency*

Bringing to Temperature of Test.

9. The temperature of the sample shall be brought to within 1° F. (0.5° C.) of 77° F. (25° C.) before the test. If the sample is initially within 3 to 4° F. (1.5 to 2° C.) of this temperature, it can be brought to 77° F. (25° C.) by placing in a water bath for 30 or 40 minutes, but if the initial temperature is outside this range it must be placed in the constant temperature bath for 1½ hours to insure reaching the final constant temperature. If the room temperature is more than 3 to 4° F. (1.5 to 2° C.) from 77° F. (25° C.) a lid should be placed on the can, sealing with grease to prevent the entrance of water and the can immersed in the bath for the required period as, otherwise, the temperature of the surface will be different from that of the main body of the grease. Any holes or seams in the can should also be sealed with grease or paraffin.

Preparation of Sample.

10. The surface of the grease shall be cut level and very smooth with a knife. Care must be taken not to work the surface of the grease. Any crust or discolored layers shall be completely removed before testing.

Procedure.

11. The can of grease shall be placed on the penetrometer table and the cone lowered until the tip just touches the surface. Watching the shadow of the tip aids in accurately setting the cone. The scale shall then be set to zero and the plunger released suddenly and kept released for five seconds. The penetration is read from the scale (the units being tenths of a millimeter) by the same procedure as is customary in measuring asphalt penetration as described in the Standard Method of Test for Penetration of Bituminous Materials

(Serial Designation: D 5) of the American Society for Testing Materials.[1]

Spacing of Tests in Cans.

12. In making tests, the total surface area disturbed by the test has a diameter about equal to the measured depth of penetration. In order to prevent one test from being affected by another disturbed area or by sides of the can, in starting a test the tip shall never be placed closer to the sides of the can or the edge of a previous hole than the penetration distance of that particular grease. The grease shall not be smoothed over for further tests.

Number of Tests Required.

13. Five tests shall be made on each sample and the average reported as the consistency, if the mean deviation of these readings does not exceed 3.0 per cent. If the mean deviation does exceed 3.0 per cent, the average of ten readings shall be reported as the consistency of the sample. More than one 1-lb. can will be needed for the required number of tests on the softer greases.

(*B*) *Procedure for Worked Consistency*

Procedure for Worked Consistency

14. The procedure for worked greases shall be the same as that for unworked consistency except that the grease to be tested shall be transferred to the worker, which shall be filled heaping full with a minimum inclusion of air, brought to within 2° F. (1° C.) of 75° F. (24° C.) and worked with 60 double strokes of the plunger.[2] The top and plunger can then be removed, the surface smoothed over and the test made at once, if the temperature is within 1° F. (0.5° C.) of 77° F. (25° C.). As soon as one test is made, the surface may be smoothed over for the next, taking care to avoid creating air pockets. If desired, the grease may be transferred carefully to another container before bringing to 77° F. (25° C.) and testing. One 1-lb. sample is sufficient for all tests.

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[1] A.S.T.M. Standards Adopted in 1925.

[2] In general quite similar results may be obtained by working the grease thoroughly for five minutes with a spatula on a flat plate, providing care is taken to prevent working in much air. For referee tests, however, the standard worker must be used.

TENTATIVE METHODS OF TEST
FOR
VISCOSITY OF PETROLEUM PRODUCTS AND LUBRICANTS[1]

Serial Designation: D 88 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1921; REVISED, 1923, 1924, 1925.

Choice of Instrument.

1. (*a*) Viscosity shall be determined by means of the Saybolt Universal or Saybolt Furol Viscosimeter.

(*b*) In general, the Saybolt Universal Viscosimeter shall be used for lubricants and the Saybolt Furol Viscosimeter for fuel oils and other oils of similar viscosity.

TABLE I.—DIMENSIONS OF OIL TUBES.

Dimensions	Saybolt Universal Viscosimeter			Saybolt Furol Viscosimeter		
	Minimum, cm.	Normal, cm.	Maximum, cm.	Minimum, cm.	Normal, cm.	Maximum, cm.
Inside diameter of outlet tube	0.1750	0.1765	0.1780	0.313	0.315	0.317
Outside diameter of outlet tube at lower end	0.28	0.30	0.32	0.40	0.43	0.46
Length of outlet tube[1]	1.215	1.225	1.235	1.215	1.225	1.235
Height of overflow rim above bottom of outlet tube[1]	12.40	12.50	12.60	12.40	12.50	12.60
Outside diameter of overflow rim, at the top[1,b]	[a]	....	3.30	[a]	....	3.30
Diameter of container[1]	2.955	2.975	2.995	2.955	2.975	2.995
Depth of cylindrical part of container[1]	8.8	....	....	8.8	....	....
Diameter of container between bottom of cylindrical part of container and top of outlet tube[1]	0.9	....	....	0.9	....	....

[1] This dimension is identical in the Saybolt Universal and the Saybolt Furol instruments.

[a] The minimum value shall preferably be not less than 3.2 cm.

[b] The section of overflow rim shall be bounded by straight lines, except that a fillet is permissible at the junction with the bottom of the gallery.

(*c*) The Saybolt Universal Viscosimeter shall not be used for times of flow less than 32 seconds.

I. APPARATUS

Viscosimeters.

2. (*a*) The Saybolt viscosimeters (see Fig. 1) are made entirely of metal. The oil tube, *A*, is fitted at the top with an overflow cup, *B*, and the tube is surrounded by a bath. At the bottom of the oil tube is a small outlet tube through which the oil to be tested flows into a receiving flask, Fig. 2, whose capacity at 20° C. (68° F.) to a mark on its neck is 60, ±0.15, cc. The outlet tube is of hard and non-

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

corrosive metal such as stainless steel, monel metal, etc. The inside diameter of neck of flask, at the level where the graduation mark is placed, is 0.8 to 1.1 cm. The lower end of the outlet tube is enclosed by a larger tube, which when stoppered by a cork, *C*, acts as a closed air chamber and prevents the flow of oil through the outlet tube until the cork is removed and the test started. A looped string may be attached to the lower end of the cork as an aid to its rapid removal. The temperatures in the oil tube and in the bath are shown by thermometers. The bath may be heated by any suitable means. The oil tube shall be thoroughly cleaned, and all oil entering the oil tube shall be strained through a 100-mesh wire strainer. A stop watch shall be used for taking the time of flow of the oil and a pipette shall be used for draining the overflow cup.

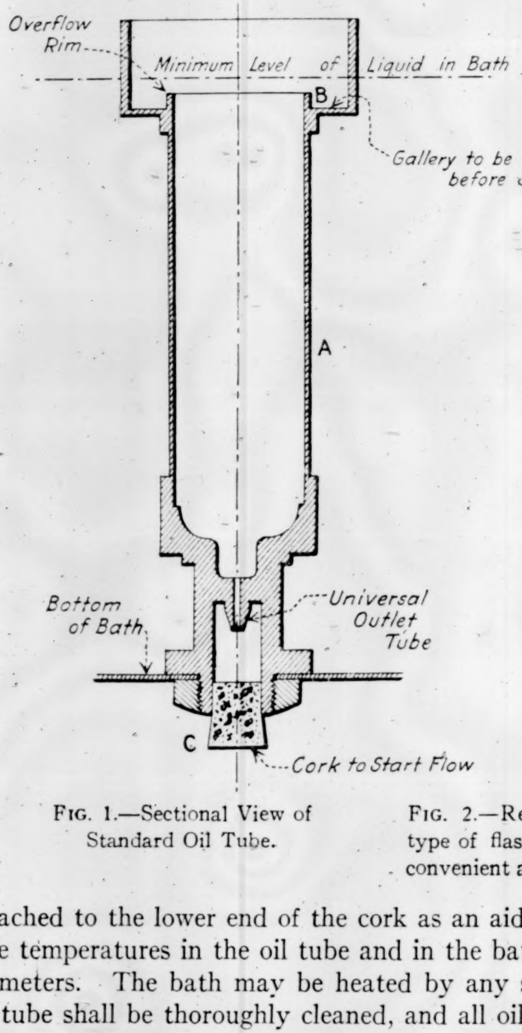
Overflow Rim
Minimum Level of Liquid in Bath
B
Gallery to be Drained before Starting Flow
A
Bottom of Bath
Universal Outlet Tube
C
Cork to Start Flow

FIG. 1.—Sectional View of Standard Oil Tube.

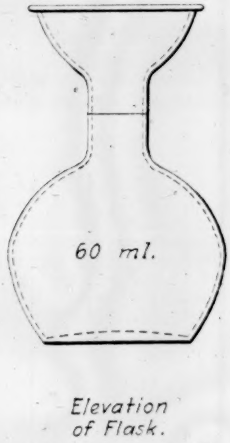
60 ml.
Elevation of Flask.

FIG. 2.—Receiving Flask. (This type of flask is recommended as convenient and durable.)

Oil Tube. (*b*) The oil tubes, which may be standardized by the U. S. Bureau of Standards, shall conform to the dimensions given in Table I. The time of flow shall be within ± 1 per cent of the time as obtained with the Bureau of Standards' master tube.[1]

Thermometers. (*c*) The bath and oil tube thermometers shall conform to the following requirements. They cover two sets of 4 thermometers each, one set being graduated in Fahrenheit degrees and the other set in Centigrade degrees, the ranges being chosen to include the temperatures commonly used in testing.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGES AND SUBDIVISIONS:

Range 66– 80° F.,	subdivision in 0.2° F.	for tests at 77° F.
" 94–108° F.,	" 0.2° F.	" 100° F.
" 120–134° F.,	" 0.2° F.	" 122 and 130° F.
" 204–218° F.,	" 0.2° F.	" 210° F.

or

Range 19–27° C.,	subdivision in 0.1° C.	for tests at 25° C.
" 34–42° C.,	" 0.1° C.	" 40° C.
" 49–57° C.,	" 0.1° C.	" 50° C.
" 95–103° C.,	" 0.1° C.	" 100° C.

TOTAL LENGTH: 252 to 256 mm. (9.92 to 10.08 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 mm. (0.236 to 0.275 in.). The stem shall preferably be made with an enlargement not less than 4 nor more than 7 mm. (0.16 to 0.28 in.) in length, having a diameter 2 to 3 mm. (0.08 to 0.12 in.) greater than that of the stem, the bottom of the enlargement being 114 mm. (4.5 in.) above the bottom of the bulb.

BULB: Corning normal or equally suitable thermometric glass.
Length, 25 to 35 mm. (1.0 to 1.4 in.).
Diameter, not less than 5 mm. (0.197 in.) and not greater than that of stem.

DISTANCE TO LOWEST SPECIFIED GRADUATION FROM BOTTOM OF BULB: 135 to 150 mm. (5.3 to 5.9 in.).

DISTANCE TO HIGHEST SPECIFIED GRADUATION FROM TOP OF STEM: 20 to 35 mm. (0.8 to 1.4 in.).

CONTRACTION CHAMBER: To be of long narrow type, top to be not more than 60 mm. (2.36 in.) above bottom of bulb, mercury to stand in contraction chamber at 32° F. (0° C.).

EXPANSION CHAMBER: To permit heating the thermometer 90° F. (50° C.) above highest temperature on scale, and in all cases to permit heating to 212° F. (100° C.).

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures and letters clear cut and distinct. The first and each succeeding 1° F. or 0.5° C. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 2° F. or 1° C.

IMMERSION: Total.

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[1] This tube represents the average mechanically perfect tube, based on the A.S.T.M. specifications, and has been approved by the American Petroleum Institute Committee on Viscosity Standards.

SPECIAL MARKING: "A.S.T.M." Viscosity test points to be numbered in full figures and in red, other graduations and figures in black. A serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale shall not exceed 0.2° F. (0.1° C.)

STANDARDIZATION: The thermometers are to be standardized for the condition of total immersion. Correction for emergent stem shall not be applied.

CASE: Each thermometer shall be supplied in a suitable case on which shall appear the marking, "A.S.T.M., Saybolt Viscosimeter Thermometer" and the range.

II. TEMPERATURE OF TESTING

Temperature.

3. (*a*) With the Saybolt Universal viscosimeter, determinations shall be made at 100° F. (37.8° C.), 130° F. (54.4° C.) or 210° F. (98.9° C.).

(*b*) With the Saybolt Furol viscosimeter, determinations shall be made at 122° F. (50° C.).

(*c*) Viscosities shall be expressed as .... seconds, Saybolt Universal (or Saybolt Furol), being the time in seconds for the delivery of 60 cc. of oil.

(*d*) Fuel oils and other oils of similar viscosity showing a time of less than 25 seconds, Saybolt Furol, at 122° F., shall be tested on the Saybolt Universal at 122° F. Oil showing a time of less than 32 seconds, Saybolt Universal, at 122° F., shall be measured in the Saybolt Universal at 100° F. (37.8° C.). These methods of test do not apply to fuels having a viscosity at 100° F. of less than 32 seconds Saybolt Universal, which are not considered to be fuel oils.

III. PROCEDURE

Procedure.

4. In tests at 100 and 130° F. (37.8 and 54.4° C.), the bath temperature, throughout the test, shall not vary more than ±0.1° F. (0.06° C.) from the predetermined temperature which will maintain thermal equilibrium until the oil tube thermometer is withdrawn. In tests at 210° F. (98.9° C.), a variation of ±0.2° F. (0.12° C.) is permitted. Water shall be used as the bath liquid at 100 and 130° F. (37.8 and 54.4° C.).[1]

Any construction of bath may be employed provided the bath temperature necessary to maintain thermal equilibrium (while the oil tube is well stirred by the oil tube thermometer) is not in excess of 100.25, 122.35, 130.50 and 212.00° F. (37.9, 50.2, 54.7 and 100.0°C.), respectively, for the standard temperatures previously mentioned. The level of the bath liquid shall not be lower than 0.5 cm. above the overflow rim of the oil tube. In tests at 210° F. (98.9° C.), the water shall be vigorously stirred, and may be heated by the direct injection of steam near the bottom of the bath. A bath of other suitable liquid may be used provided it is properly heated and stirred.[1] The heating and stirring of the bath for other temperatures may be accomplished by any suitable means, provided the source of heat is not less than the following distances from any part of the oil tube: 2 in. (5 cm.) with an external heater, $1\frac{1}{4}$ in. (3 cm.) with an immersion heater. Viscosity determinations shall be made in a room free from draughts, and from rapid changes in temperature. The room temperature shall be between 68 and 86° F. (20 and 30° C.).[2] All oil introduced into the oil tube either for cleaning or for test shall first be passed through the strainer.

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[1] In ordinary routine testing it is frequently desirable to employ oil as a bath medium instead of water. This is allowable provided the temperature of the oil bath is adjusted so that the necessary condition of thermal equilibrium is maintained. It is usually necessary to maintain the oil bath at slightly higher temperatures than are necessary when water is the bath medium. Oil bath temperatures will need to be from 0.1 to 0.2° F. (0.06 to 0.11° C.) higher for tests at 100° F. (37.8° C.) and from 1.5 to 2.0° F. (0.83 to 1.11° C.) higher for tests at 210° F. (98.9° C.) than the corresponding water bath temperatures.

To make the test, heat the oil to the necessary temperature and clean out the oil tube. Pour some of the oil to be tested through the cleaned tube. Insert the cork stopper into the lower end of the air chamber at the bottom of the oil tube, sufficiently to prevent the escape of air, but not to touch the small outlet tube.

Heat the oil to be tested, outside the viscosimeter, to slightly but not more than 3° F. (1.7° C.) above the temperature at which the viscosity is to be determined, and pour it into the oil tube until it ceases to overflow into the overflow cup. By means of the oil-tube thermometer, keep the oil in the oil tube well stirred and also stir well the liquid in the bath. It is extremely important that the temperature of the bath be maintained constant during the entire time consumed in making the test. When the temperature of the bath and of the oil in the oil tube are constant and the oil in the oil tube is at the desired temperature, withdraw the oil tube thermometer; quickly remove the surplus oil from the overflow cup by means of a pipette so that the level of the oil in the overflow cup is below the level of the oil in the tube proper; place the 60-cc. flask, Fig. 2, in position so that the stream of oil from the outlet tube will strike the neck of the flask so as to avoid foam. Snap the cork from its position, and at the same instant start the stop watch. Stir the liquid in the bath during the run and carefully maintain it at the previously determined proper temperature. Stop the watch when the bottom of the meniscus of the oil reaches the mark on the neck of the receiving flask.

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[1] For example, the bath liquid may be water to which glycerin or salt has been added.

[2] These limits are necessary for extreme accuracy in standardization and referee tests, but for routine purposes the use of higher temperatures up to 100° F. (37.8° C.) will not ordinarily cause an error of more than 1 per cent.

The time in seconds for the delivery of 60 cc. of oil is the Saybolt Universal (or Saybolt Furol) viscosity of the oil at the temperature at which the test was made.

With proper attention to details of method of procedure, duplicate results should not differ from each other by more than 1 per cent.

TENTATIVE METHOD OF TEST
FOR
COLOR OF PETROLATUM BY MEANS OF THE UNION COLORIMETER[1]

Serial Designation: D 218 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. **It is not a Standard of the Society and is subject to annual revision.**

ISSUED, 1925

I. APPARATUS

Scope. 1. The color of all petrolatums shall be determined by means of the Union Colorimeter.

Apparatus. 2. (*a*) The Union Colorimeter, Fig. 1, shall consist of a box, *a*, glass color standards, a glass jar, a light shield, *b*, and a daylight lamp, *c*.

(*b*) The box, *a*, Fig. 1, shall have an interior finish of dull black, and may be constructed of metal or wood, approximately 18 in. in length and $1\frac{1}{2}$ in. in depth in inside dimensions, the observation end being $2\frac{1}{8}$ in. in width and the end next to the source of light 3 in. in width.

A transverse vertical partition having a circular hole in the end $\frac{1}{2}$ in. in diameter shall be placed $1\frac{1}{8}$ in. from the observation end of the box. In the opposite end shall be mounted a rectangular piece of pure white opal glass, approximately $3\frac{1}{4}$ by $1\frac{3}{4}$ by $\frac{1}{16}$ in., which shall be ground or etched on the inner side to diffuse light rays. Approximately $2\frac{1}{8}$ in. from the light end of the box shall be a second vertical partition 1 in. in thickness having two openings about $\frac{3}{4}$ in. in diameter at the light-source side and about $\frac{9}{16}$ in. in diameter on the opposite side, symmetrically located in regard to the vision line. In case the standards are not mounted, a vertical partition shall be placed approximately $3\frac{1}{4}$ in. from the light-source end, having a thickness of $\frac{1}{8}$ in., and shall have two circular openings $\frac{9}{16}$ in. in diameter and $1\frac{1}{2}$ in. apart. Extending from the opal glass to this partition along the horizontal axis of the box shall be a vertical partition approximately

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

$\frac{1}{8}$ in. in thickness for a distance of $1\frac{5}{8}$ in., and then continuing with a thickness of $\frac{7}{8}$ in. until abutting against the transverse partition, which in conjunction serves to divide the light end of the box into two compartments. In the top of the box, immediately over the centers of these two compartments, there shall be circular holes of such a size to accommodate the standard jar and 4-oz. oil sample bottles. In the top of the box, directly above the $\frac{3}{4}$-in. opening in the left side of the second partition, there shall be an opening or slot suitable for placing the glass color standards in a vertical position in the opening in the partition. In case the color standards are not mounted in a magazine, this opening shall be provided with a sliding cover so that all light may be excluded. In case all the glass standards are mounted in a magazine, suitable arrangements shall be made for this magazine to be raised or lowered through the opening provided for the glass standards, and when such a magazine is used, a hole may be located in the left side of the box through which the numbers of the mounted glass standards may be read. For convenience, numbers may be

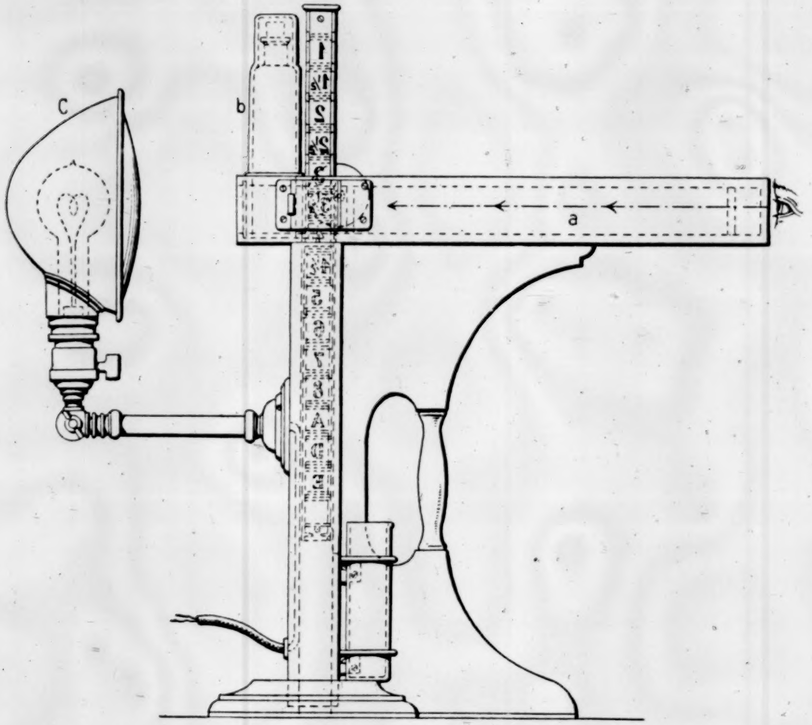

FIG. 1.—Union Colorimeter.

stamped on the magazine as mirror images, and a swinging mirror may be provided on the side of the box so that the operator may read the numbers on the magazine from the front of the instrument.

(*c*) The glass color standards shall be 15 in number as specified in Table I. These glasses may be kept separately or for convenience may be mounted in a magazine. If so mounted, there shall be a blank opening provided in the magazine so that a bottle of petrolatum may be compared in the colorimeter with a bottle of oil or with a second bottle of petrolatum as well as a sample of petrolatum and the glass color standards. The glass color standards shall be not less than $\frac{5}{8}$ in. across.

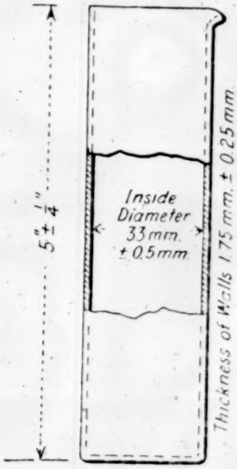

FIG. 2.—Standard Glass Jar.

(*d*) The standard glass jar, Fig. 2, shall be a true cylinder of clear colorless glass. The internal diameter shall be not less than 32.5 mm. nor more than 33.5 mm. The thickness of the wall shall be 1.75 mm. ±0.25 mm. The height of the cylinder shall be 5 in. ±$\frac{1}{4}$ in.

(*e*) The light proof cover, *b*, shall be of any suitable material. It shall be dull black on the inside and of such size as to completely shield all light from the tops of the cylinders, or oil sample bottles, when they are in place in the colorimeter.

(*f*) The artificial daylight lamp, *c* (Fig. 1), may for convenience be fastened to the base supporting the box. It shall consist of a 50-watt *C2* national mazda lamp, fitting into a socket on a swivel fitted with a parabolic metal reflector. The inside of this reflector shall be brightly silvered at all times with bright aluminum paint or brightly polished silver or nickel. In the front of the reflector shall be mounted a dished, but not lenticular, glass color screen of such composition and color as, in conjunction with the *C2* mazda lamp, will yield, by spectrum analysis, a light closely approximating northern daylight. When in normal position, the color screen shall be 4 in. from the white opal glass.

In case electric current is not available, the colorimeter may be used by exposing the opal glass to northern daylight with no colored objects in the immediate foreground, but the artificial daylight lamp shall be required in referee work.

Table I.—Glass Color Standards of Union Colorimeter.

A.S.T.M. Color Numbers	Union Petroleum Co. Designation	National Petroleum Association Color Numbers (1915)	National Petroleum Association Names	Lovibond Analysis		
				Red 200 Series	Yellow 510 Series	Blue 1180 Series
1	G	No. 1 N. P. A	Lily White	0.12	2.4	....
1½	H	No. 1½ N. P. A	Cream White	0.60	8.0	....
2	I	No. 2 N. P. A	Extra Pale	2.5	26.0	....
2½	J	No. 2½ N. P. A	Extra Lemon Pale	4.6	27.0	....
3	K	No. 3 N. P. A	Lemon Pale	6.9	32.0	....
3½	L	[a]	Extra Orange Pale	9.4	45.0	....
4	M	No. 4 N. P. A	Orange Pale	14.0	50.0	0.55
4½	N	No. 4½ N. P. A	Pale	21.0	56.0	0.55
5	O	No. 5 N. P. A	Light Red	35.0	93.0	....
6	P	No. 6 N. P. A	Dark Red	60.0	60.0	0.55
7	Q	Q	Claret Red	60.0	106.0	1.8
8	R	[b]		166.0	64.0	....
A	A	Cylinder Oil	Extra Light Filtered	10.2	29.0	....
D	D	Cylinder Oil	Light Filtered	21.0	31.0	....
E	E	Cylinder Oil	Medium Filtered	89.0	56.0	....

[a] N. P. A. Color of 1915 changed slightly to make 3½ more nearly midway between 3 and 4.

[b] A new color.

II. PROCEDURE

3. The petrolatum shall be heated to 20 to 30° F. (11 to 17° C.) above the melting point. If darker than No. 8 color (Table I) it shall be diluted with water white kerosine, the color of which is not darker than No. 21 as determined in accordance with the Tentative Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (Serial Designation: D 156 – 23 T) of the American Society for Testing Materials,[1] a mixture of exactly 15 per cent by volume of petrolatum and 85 per cent by volume of kerosine being used in all cases.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 682 (1923); also 1925 Book of A.S.T.M. Tentative Standards, p. 374.

The water white kerosine shall be heated to a temperature 20 to 30° F. (11 to 17° C.) above the melting point of the petrolatum being tested before mixing them together.

This mixture shall then be placed in the standard glass jar and compared with the three cylinder oil standards or optionally with the standards ranging from No. 1 to No. 8, inclusive.

The daylight lamp may be brought closer to the opal glass in determining the darker colors. The numbers or letters designating the color of all petrolatum that has been diluted to make determinations shall be followed by the designation "Dil."

TENTATIVE METHOD OF TEST
FOR
BURNING QUALITY OF LONG-TIME BURNING OIL FOR RAILWAY USE[1]

Serial Designation: D 219 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. The burning test for long-time burning oil is intended for the determination of the burning quality of special kerosine oil used in railway semaphore signal lamps.

I. APPARATUS

Lamp. 2. The lamp shall be the standard American Railway Association, Signal Section, semaphore lamp.

Fount. 3. A 31 fluid ounce fount, about 3 in. high, measured from inside the bottom to top of burner collar, and fitting the standard lamp, shall be used.

Burner. 4. The burner shall be a *Dressel No. 23 Long-Time Burner*, New York Central Type, or any other burner meeting the following requirements: A nickel plated long-time burner made of suitable weight metal, provided with the conventional wick adjusting device, with any convenient means of fitting to fount, and in other respects corresponding to the following requirements:

> *Flame Spreader.*—Cylindrical, $\frac{5}{16}$ in. in height and $\frac{1}{2}$ in. in inside diameter, soldered to the top of wick tube. Two V-shaped slots, placed opposite to each other, with top of V $\frac{1}{4}$ in. wide and bottom ending just above top of wick tube.
>
> *Wick Tube.*—$1\frac{15}{16}$ in. in length, $\frac{1}{4}$ in. in inside diameter, and extending $\frac{5}{8}$ in. above top of wick tube.
>
> *Chimney Support Ring.*—$1\frac{1}{4}$ in. in inside diameter, $\frac{7}{16}$ in. below top of flame spreader, equipped with 4 springs to hold chimney in place.
>
> *Air Vents.*—Four $\frac{1}{16}$ in. holes placed symmetrically in flame spreader just above the top of the wick tube; four rectangular

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

openings, $\frac{7}{16}$ in. high, by $\frac{9}{16}$ in. wide, placed below the chimney support ring and between the chimney springs.

Chimney.

5. The chimney shall be a Macbeth No. 55 D Chimney, or any other chimney meeting the following requirements: Straight wall glass $2\frac{1}{2}$ in. high and $\frac{1}{8}$ in. in inside diameter, with rim at top and bottom.

Wick.

6. A new gray round felt wick, approximately $7\frac{3}{4}$ in. long and $\frac{3}{16}$ in. in diameter, made by sewing a strip of felt $\frac{1}{2}$ in. wide by $7\frac{3}{4}$ in. long by $\frac{1}{8}$ in. thick into proper form is suitable. The wick shall be washed with redistilled ether and dried at room temperature before use.

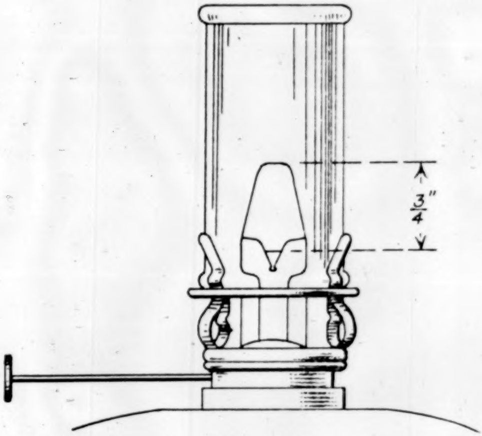

FIG. 1.

Balance.

7. A platform balance sensitive to 1 g. shall be used.

Room.

8. The lamp may be burned in any well ventilated room, or out of doors, if not subjected to the direct rays of the sun.

Sight Gage.

9. A sight gage, or other suitable flame measuring device, accurate to $\frac{1}{16}$ in. shall be used. The sight gage shown in Fig. 2 is satisfactory.

II. PROCEDURE

Procedure.

10. Seven hundred and fifty cubic centimeters of the oil to be tested shall be placed in the fount. The burner, wick, chimney and fount shall be assembled, the rough edge trimmed from the wick and the wick centered in the flame spreader, lighted and adjusted to give a flame $\frac{3}{4}$ in. high, measured from top of wick. This, and all following flame measurements, should be made with the assembly inside of the lamp, with slide open, and in a room free from drafts. The flame should have a flat top and be of the general shape indicated

in Fig. 1. If this cannot be accomplished by leveling the lamp, the burner should be rejected. Inability to secure the specified flame shape will be due, in such cases, to improper alignment of the sides of the flame spreader. It is essential that the specified flame shape be adhered to, as any deviation from it may influence the rate of oil consumption and the flame drop. The assembly shall be weighed while burning, then placed in the lamp. The wick shall be readjusted, if necessary, without removing the fount from the lamp, during the first four hours. The lamp shall be allowed to burn continuously for the duration of the test. After 96 hours, the fount assembly shall be removed from the lamp, weighed while burning and then replaced in the lamp. After 120 hours the lamp shall again be weighed and the average rate of oil consumption per hour calculated for the 96 to

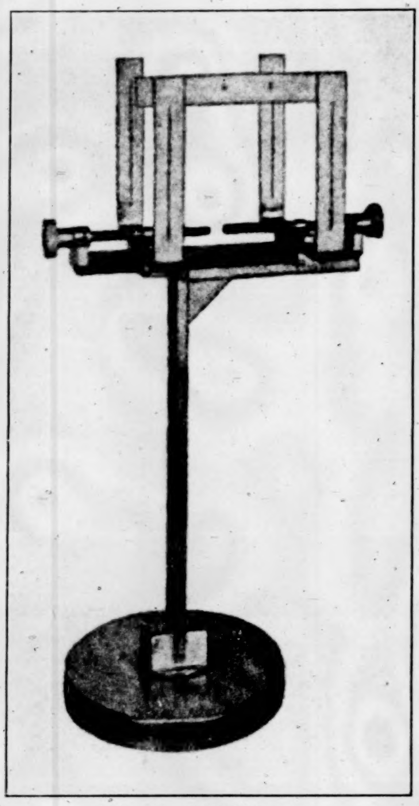

FIG. 2.—Sight Gage.

120-hour period. From this rate and the original weight of oil in the fount, the number of additional hours required to make the total quantity of oil consumed 650 cc. shall be calculated and the lamp burned the calculated length of time. The oil consumption shall be verified by weighing at the end of the test. The time required to burn 650 cc. will, in general, be not less than 120 hours and not more than 144 hours if proper attention is paid to initial flame height and shape. At the end of the test, observe and record the change in height of flame, and condition of the wick and chimney.

TENTATIVE METHOD OF TEST
FOR
NEUTRALIZATION NUMBER OF PETROLEUM PRODUCTS AND LUBRICANTS[1]

Serial Designation: D 188 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. The methods described herein are designed to indicate in petroleum products and lubricants and compounded products the presence of organic constitutents having acid characteristics and the contamination by alkalies and mineral acids.

Definitions. 2. (*a*) The Neutralization Number is the weight in milligrams of potassium hydroxide required to neutralize one gram of oil.

NOTE.—The Neutralization Number expresses the total amount of the mineral acid, and of the organic constituents having acid characteristics.

(*b*) The Alkali Neutralization Number is defined as the weight of acid required to neutralize one gram of oil, expressed in equivalent milligrams of potassium hydroxide.

(*c*) The Mineral Acid Neutralization Number is the number of milligrams of potassium hydroxide required to neutralize the mineral acid content in one gram of oil.

SOLUTIONS REQUIRED

3. (*a*) *Aqueous Potassium Hydroxide* (1 cc. = 5 mg. KOH).—Dissolve 5.1 g. of potassium hydroxide, c.p., in one liter of freshly boiled and cooled distilled water. Add a very small amount of barium hydroxide, sufficient to precipitate any potassium carbonate present. Standardize this solution against Bureau of Standards certified benzoic acid, using phenolphthalein as an indicator according to the relation:

$$\frac{5 \text{ mg. KOH}}{x \text{ mg. benzoic acid}} = \frac{56.104 \text{ g. KOH}}{122.048 \text{ g. benzoic acid}}$$

$$1 \text{ cc. of KOH} = 10.88 \text{ mg. benzoic acid.}$$

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

This weight of benzoic acid is required for standardization.

Make necessary adjustments so that the value of potassium hydroxide equals 5 mg. KOH per 1 cc.

NOTE.—Fit the solution bottle with a guard tube of soda lime to prevent access of carbon dioxide. The solution should be standardized at necessary intervals.

The weight of benzoic acid should be dissolved in 50 cc. of 95-per-cent alcohol and titrated cold. For blank, use same amount of alcohol and correct the titration.

(*b*) *Sulfuric Acid Solution* (1 cc. = 5 mg. KOH).—Match a sulfuric acid solution so that 1 cc. of the acid will be required to neutralize 1 cc. of the KOH, using phenolphthalein as an indicator, titrating in boiling solution.

(*c*) *Neutralized 95-per-cent Alcohol.*—Add a few drops of phenolphthalein and neutralize carefully the alcohol to a very faint pink end point with some of the above prepared alkali solution.

(*d*) *Phenolphthalein Indicator.*—Dissolve 10 g. of the indicator in 1 liter of 95-per-cent alcohol, preferably ethyl. Use 1 cc. of this strength for titration.

(*e*) *Methyl Orange Indicator.*—Dissolve 1 g. of the indicator in 1 liter of distilled water. Use 0.1 cc. of this strength for titration.

METHODS

4. *Method A.—Neutralization Number of Petroleum Products.*

Weight of Oil: Approximately 20 g. weighed accurately.

Volume of Solvent: 100 cc. of a mixture of 1:1 neutralized alcohol and distilled water.

Procedure: Agitate oil and solvent thoroughly and heat to boiling. Add 1 cc. of phenolphthalein indicator and titrate rapidly, with vigorous agitation, to a sharp pink end point. The titration must be completed in a hot solution, reheating same if found necessary.

The color change is noted in the alcohol water layer.

Calculation:

$$\frac{(\text{Cubic centimeters of KOH}) \times 5}{\text{Weight of oil taken}} = \text{mg. KOH per 1 g. of oil.}$$

5. *Method B.—Neutralization Number of Compounded Petroleum Products.*

Weight of Oil: Approximately 10 g. weighed accurately.

Volume of Solvent: 50 cc. of neutralized alcohol.

Procedure: Agitate oil and solvent thoroughly and heat to boiling. Add 1 cc. of phenolphthalein indicator and titrate rapidly, with vigorous agitation, to a sharp pink end point. The titration must be completed in a hot solution, reheating same if found necessary.

The color change is noted in the alcohol layer.

6. *Method C.—Alkali or Mineral Acid Neutralization Number in Non-compounded and compounded petroleum products.*

Weight of Oil: 25 to 50 g. weighed accurately.

Volume of Solvent: Boiling distilled water, 200 cc.

Procedure: Pour the oil and water in a 500-cc. separatory funnel. Shake vigorously and after oil and water have separated, drain water into a 500-cc. casserole. Add 100 cc. of boiling water to oil and agitate. Drain separated water to casserole. Repeat washing and to the accumulated 100 cc. of water add 1 drop of phenolphthalein. Boil. If solution turns pink add 1 cc. of the indicator and titrate with the sulfuric acid solution until extraction is colorless.

If the addition of 1 drop of phenolphthalein causes no change in color, add 0.1 cc. of methyl orange. If color changes to red or pink, titrate with the KOH solution until the red color is just discharged.

An equal volume of distilled water shall be titrated with the standard solution used, and the amount of alkali or acid required shall be subtracted from the titration for blank correction.

Calculation: If the acid solution was required to discharge the phenolphthalein end point, the alkali neutralization number equals:

$$\frac{(\text{Cubic centimeters of acid}) \times 5}{\text{Weight of oil}}$$

If the alkali solution was required to discharge the methyl orange end point, the mineral acid neutralization number equals:

$$\frac{(\text{Cubic centimeters of alkali}) \times 5}{\text{Weight of oil}}$$

TENTATIVE METHOD OF TEST
FOR
CLOUD AND POUR POINTS OF PETROLEUM PRODUCTS[1]

Serial Designation: D 97 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921; Revised, 1922, 1923, 1925.

Cloud Point. 1. (*a*) The Cloud Point of a petroleum oil is that temperature at which paraffin wax or other solid substances begin to crystallize out or separate from solution when the oil is chilled under certain definite specified conditions.

Pour Point. (*b*) The Pour Point of a petroleum oil is the lowest temperature at which this oil will pour or flow when it is chilled without disturbance under certain definite specified conditions.

Scope. 2. (*a*) The test for cloud point shall be used only for oils which are transparent in layers 1½ in. thick.

(*b*) The test for pour point shall be used for all other petroleum oils and may be used for oils on which the test for cloud point is permitted.

I. APPARATUS

(See Fig. 1)

Test Jar. 3. The test jar, *a*, shall be of clear glass, cylindrical form, flat bottom, approximately 1¼ in. in inside diameter and 4½ to 5 in. high. An ordinary 4-oz. oil sample bottle may be used if the test jar is not available.

Thermometers. 4. The thermometer, *b*, shall conform to the requirements of one of the following specifications:

(*a*) These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −38 to +50° C. or −36 to +120° F., respectively.

Type: Etched stem, glass.

Liquid: Mercury.

Range and Subdivision: −38 to +50° C. in 1° C. or −36 to +120° F. in 2° F.

Total Length: 220 to 224 mm. (8.69 to 8.81 in.).

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter, 7.0 to 8.0 mm. (0.28 to 0.31 in.).

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

BULB: Corning normal or equally suitable thermometric glass.
Length, not over 9.5 mm. (0.37 in.).
Diameter, not greater than stem.

DISTANCE TO −38° C. OR −36° F. LINE FROM BOTTOM OF BULB: 120 to 130 mm. (4.73 to 5.12 in.).

DISTANCE TO +49° C. OR +120° F. LINE FROM TOP OF THERMOMETER: 19 to 25 mm. (0.75 to 0.98 in.).

EXPANSION CHAMBER: To permit heating to 100° C. or 212° F.

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Plain.

GRADUATION: All lines, figures, and letters clear cut and distinct. Lines at multiples of 5° C. or 10° F. to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

IMMERSION: 108 mm. or 4¼ in. The words "108-mm. immersion" on Centigrade thermometers or "4¼-in. immersion" on Fahrenheit thermometers and a line around the stem 108.0 mm. or 4.25 in. above the bottom of the bulb shall be etched on the thermometer.

SPECIAL MARKING: "A.S.T.M. Cloud and Pour," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale, when the thermometer is standardized as provided below, shall not exceed 0.5° C. or 1° F.

STANDARDIZATION: The thermometer shall be standardized at the ice point and at intervals of approximately 20° C. or 40° F. for 108-mm. or 4¼-in. immersion and for an average temperature of 21° C. or 70° F. for the emergent mercury column.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M.Cloud and Pour, −38 to +50° C." or "A.S.T.M. Cloud and Pour, −36 to +120° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

(*b*) These specifications cover a special low cloud and pour test thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −60 to +20° C. or −70 to +70° F.

TYPE: Etched stem, glass.

LIQUID: Toluene or other suitable liquid colored red with a permanent dye.

RANGE AND SUB-DIVISION: −60 to +20° C. in 1° C. or −70 to +70° F. in 2° F.

TOTAL LENGTH: 227 to 231 mm. (8.92 to 9.08 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter 7.0 to 8.0 mm. (0.28 to 0.31 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 8.0 to 9.5 mm. (0.31 to 0.37 in.).
Diameter, 5.0 to 6.5 mm. (0.22 to 0.26 in.).

DISTANCE TO −57° C. or −70° F. LINE FROM BOTTOM OF BULB: 120 to 130 mm. (4.73 to 5.12 in.).

DISTANCE TO +20° C. or +68° F. LINE FROM TOP OF THERMOMETER: 35 to 45 mm. (1.38 to 1.77 in.).

EXPANSION CHAMBER: To permit heating to +60° C. or +140° F.

FILLING ABOVE LIQUID: Gas under pressure.

TOP FINISH: Plain.

GRADUATION: All lines, figures and letters clear cut and distinct, lines at multiples of 5° C. or 10° F. to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

IMMERSION: 108 mm. (4¼ in.). The words "108-mm. immersion" on Centigrade thermometers, or "4¼-in. immersion" on Fahrenheit thermometers and a line around the stem 108 mm. (4.25 in.) above the bottom of the bulb, shall be etched on the thermometer.

SPECIAL MARKING: "A.S.T.M. Low Cloud and Pour" a serial number and the manufacturer's name or trade mark shall be etched on the thermometer.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below, shall not exceed 1° C. or 2° F.

STANDARDIZATION: The thermometer shall be standardized at the ice point and at intervals of approximately 20° C. or 35° F. for 108-mm. or 4¼-in. immersion, and for an avergae temperature of 21° C. or 70° F. for the emergent liquid column.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking "A.S.T.M. Low Cloud and Pour, −60 to +20° C." or A.S.T.M. Low Cloud and Pour, −70 to +70° F.," according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications, the following definitions apply:

The total length is the overall length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

5. The cork, *c*, shall fit the test jar, and shall be bored centrally to take the test thermometer. **Cork.**

6. The jacket, *d*, shall be of glass or metal, shall be water tight, of cylindrical form, flat bottom, about 4½ in. deep, with inside diameter ½ in. greater than outside diameter of the test jar. **Jacket.**

7. A disk of cork or felt, *e*, ¼ in. thick and of the same diameter as the inside of the jacket will be required. **Cork Disk.**

8. The ring gasket, *f*, shall be about 3/16 in. thick, and made to fit snugly around the outside of the test jar and loosely inside the jacket. This gasket may be made of cork, felt or other suitable material, elastic enough to cling to the test jar and hard enough to hold its shape. The purpose of the ring gasket is to prevent the test jar from touching the jacket. **Gasket.**

9. The cooling bath, *g*, shall be of a type suitable for obtaining the required temperatures. The size and shape of the bath are optional but a support, suitable for holding the jacket firmly in a **Bath.**

vertical position, is essential. For determination of very low pour points, a smaller insulated cooling bath may be used and the test jar placed directly in it. The required bath temperatures may be maintained by refrigeration if available, otherwise by suitable freezing mixtures.

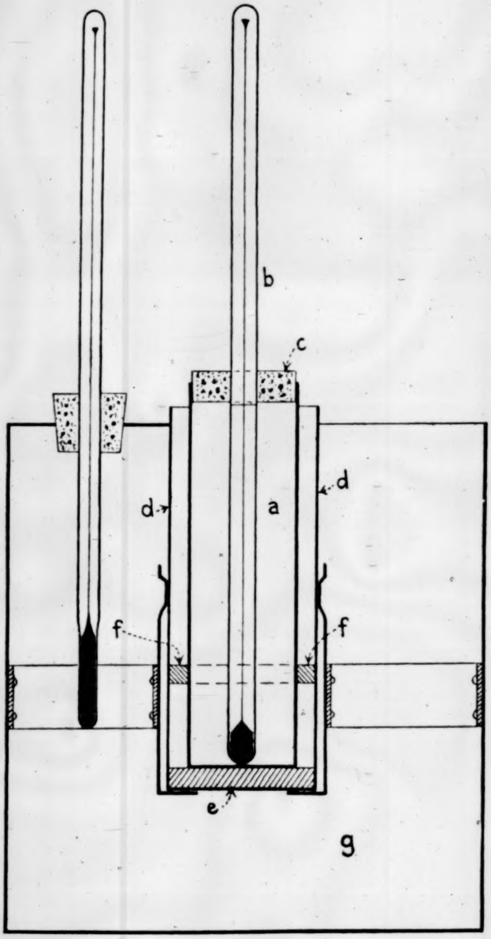

FIG. 1.—Apparatus for Cloud and Pour Test (as assembled for Cloud Test).

NOTE.—The freezing mixtures commonly used are as follows:

For temperatures down to 50° F., ice and water.
" " " " 10° F., crushed ice and sodium chloride.
" " " " −15° F., crushed ice and calcium chloride.
" " " " −70° F., solid carbon dioxide and acetone or gasoline.

The last named mixture may be made as follows: In a covered metal beaker chill a suitable amount of acetone to 10° F., or lower, by means of an ice-salt mixture. Invert a cylinder of liquid carbon dioxide and draw off carefully into a chamois skin bag the desired amount of carbon dioxide, which through rapid evaporation will quickly become solid. Then add to the chilled acetone enough of the solid carbon dioxide to give the desired temperature.

II. PROCEDURE

Procedure for Cloud Point.

10. The oil to be tested shall be brought to a temperature at least 25° F. above the approximate cloud point. Moisture, if present, shall be removed by any suitable method, as by filtration through dry filter paper until the oil is perfectly clear, but such filtration shall be made at a temperature at least 25° F. above the approximate cloud point.

The clear oil shall be poured into the test jar, *a*, to a height of not less than 2 nor more than 2¼ in. The test jar may be marked to indicate the proper level.

The test jar shall be tightly closed by the cork, *c*, carrying the test thermometer, *b*, in a vertical position in the center of the jar with the thermometer bulb resting on the bottom of the jar.

The disk, *e*, shall be placed in the bottom of the jacket, *d*, and the test jar with the ring gasket, *f*, 1 in. above the bottom shall be inserted into the jacket. The disk, jacket and inside of jacket shall be clean and dry.

The temperature of the cooling bath, *g*, shall be adjusted so that it is below the cloud point of the oil by not less than 15 nor more than 30° F. and this temperature shall be maintained throughout the test. The jacket, containing the test jar, shall be supported firmly in a vertical position in the cooling bath so that not more than 1 in. of the jacket projects out of the cooling medium.

At each test thermometer reading which is a multiple of 2° F., the test jar shall be removed from the jacket, quickly but without disturbing the oil, inspected for cloud, and replaced in the jacket. This complete operation shall require not more than three seconds.

When such inspection first reveals a distinct cloudiness or haze in the oil at the bottom of the test jar, the reading of the test thermometer, corrected for error if necessary, shall be recorded as the cloud point.

11. The oil shall be poured into the test jar, *a*, to a height of not less than 2 nor more than 2¼ in. When necessary, the oil shall be heated in a water bath just sufficiently for pouring into the test jar.

The test jar shall be tightly closed by the cork, *c*, carrying the test thermometer, *b*, in a vertical position in the center of the jar with the thermometer bulb immersed so that the beginning of the capillary shall be $\frac{1}{8}$ in. below the surface of the oil.

Heat without stirring to a temperature of 115° F. in a bath maintained at not higher than 118° F. The oil shall then be cooled to 90° F. in air or in a water bath approximately 77° F. in temperature. Oils with which the low cloud and pour test thermometer can be used from the beginning of the test shall be cooled to 60° F. in any convenient manner before placing that thermometer in position.

The disk, *e*, shall be placed in the bottom of the jacket, *d*, and the test jar, with the ring gasket, *f*, 1 in. above the bottom, shall be inserted into the jacket. The disk, gasket and inside of jacket shall be clean and dry.

After the oil has cooled enough to allow the formation of paraffin wax crystals, great care shall be taken not to disturb the mass of the oil nor to permit the thermometer to shift in the oil. Any disturbance of the spongy network of wax crystals will lead to low and fictitious results.

The temperature of the cooling bath, *g*, shall be adjusted so that it is below the pour point of the oil by not less than 15 nor more than 30° F. and this temperature shall be maintained throughout the test. The jacket, containing the test jar, shall be supported firmly in a vertical position in the cooling bath so that not more than 1 in. of the jacket projects out of the cooling medium.

Beginning at a temperature 20° F. before the expected pour point, at each test thermometer reading which is a multiple of 5° F., the test jar shall be removed from the jacket carefully and shall be tilted just sufficiently to ascertain whether there is a movement of the oil in the test jar. The complete operation of removal and replacement shall require not more than three seconds. As soon as the oil in the test jar does not flow when the jar is tilted, the test jar shall be held in a horizontal position for exactly five seconds, as noted by a stop watch or other accurate timing device and observed carefully. If the oil shows any movement under these conditions, the test jar shall be immediately replaced in the jacket and the same procedure repeated at the next temperature reading 5° F. lower.

The test shall be continued in this manner until a point is reached at which the oil in the test jar shows no movement when the test jar is held in a horizontal position for exactly five seconds. Certain lubricating oils tend to move as a whole and should be very closely observed. The reading of the test thermometer at this temperature, corrected for error if necessary, shall be recorded. The pour point shall be taken as the temperature 5° F. above this solid point.

Special Procedure for Black Oils and Cylinder Stocks

12. (*a*) In those cases where it is known that a sample has been subjected to some temperature higher than 115° F. during the preceding 24 hours, or where the history of the sample in this respect is not known, the sample shall be held in the laboratory 24 hours before testing, unless threee consecutive tests of the same sample in the same test jar check results. For these particular oils this shall be called the maximum pour point.

(*b*) The minimum pour point shall be determined by heating a sample with stirring to 220° F. The oil shall then be poured in the test jar, cooled to 90° F. as before, and the pour point determined as described in Section 11.

(*c*) The pour point of such oils shall be reported as lying between the maximum and minimum pour points (*e. g.*, Pour Point 35°/50° F.).

TENTATIVE METHODS OF TESTING GAS OILS

(GRAVITY, DISTILLATION, SULFUR, CARBON RESIDUE, POUR POINT, VISCOSITY, WATER)[1]

Serial Designation: D 158 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923; REVISED, 1925.

GRAVITY

Gravity. 1. The gravity shall be determined at 60°/60° F. (15.6°/15.6° C.) in a pyknometer, accurately standardized, or by means of a hydrometer or Westphal balance, the accuracy of which has been verified. The gravity shall be recorded as specific gravity or gravity, degrees A.P.I., at 60°/60° F. (15.6°/15.6° C.).

DISTILLATION

APPARATUS

Flask. 2. *Flask.*—The distillation flask shall be a Saybolt distilling flask, the dimensions and permissible variations being as follows:

	CENTIMETERS	INCHES	TOLERANCES, CM.
Diameter of bulb, outside	8.57	3.37	0.3
Diameter of neck, inside	1.50	0.59	0.1
Over all length, neck plus bulb	21.50	8.46	0.3
Length of vapor tube	17.50	6.89	0.5
Diameter of vapor tube, outside	0.60	0.24	0.05
Diameter of vapor tube, inside	0.40	0.16	0.05
Thickness of vapor tube wall	0.10	0.04	0.02

The center of the vapor tube shall be 6.5 cm. (2.56 in.), ±0.3 cm., below the top of the neck. The tube is approximately in the middle of the neck and set at an angle of 75 deg., ±3 deg., with the vertical.

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

3. *Condenser.*—The condenser[1] shall consist of a $\frac{9}{16}$-in. (14.29-mm.), outside diameter, No. 20 Stubbs Gage seamless brass tube, 22 in. (55.88 cm.) in length. It shall be set at an angle of 75 deg. with the vertical and shall be surrounded with a bath 15 in. (38.1 cm.) in length, approximately 4 in. (10.16 cm.) in width by 6 in. (15.24 cm.) in height. The bath shall be provided with suitable connections for circulating water through it. The lower end of the condenser tube shall be cut off at an acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the receiving graduate at a point approximately 1 to $1\frac{1}{4}$ in. (2.54 to 3.18 cm.) below the top of the graduate when it is in position to receive the distillate. **Condenser.**

4. *Shield.*—The shield[2] shall be made of approximately No. 22 gage sheet metal and shall be 19 in. (48.26 cm.) in height, 11 in. (27.94 cm.) in length and 8 in. (20.32 cm.) in width, with a door on one narrow side, with two openings, 1 in. (2.54 cm.) in diameter, equally spaced, in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings shall be $8\frac{1}{2}$ in. (21.59 cm.) below the top of the shield. There shall also be three $\frac{1}{2}$-in. (1.27-cm.) holes in each of the four sides with their centers 1 in. (2.54 cm.) above the base of the shield. **Shield.**

5. *Ring Support and Hard Asbestos Boards.*—The ring support may be of the ordinary laboratory type, 4 in. (10.16 cm.) or larger in diameter, and shall be supported on a stand inside the shield. There shall be two hard asbestos boards: One 6 by 6 by $\frac{1}{4}$ in. (15.24 by 15.24 cm. by 6.35 mm.) with a hole $2\frac{3}{4}$ in. (6.99 cm.) in diameter in its center, the sides of which shall be perpendicular to the surface; the other, an asbestos board to fit tightly inside the shield, with an opening 4 in. (10.16 cm.) in diameter concentric with the ring support. These shall be arranged as follows: The second asbestos board shall be placed on the ring and the first or smaller asbestos board on top so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat shall be applied to the flask only through the $2\frac{3}{4}$-in. (6.99-cm.) opening in the first asbestos board. **Support.**

6. *Gas Burner.*—The burner shall be so constructed that sufficient heat can be obtained to distill the product at the uniform rate **Heater.**

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[1] A condenser conforming to these specifications is illustrated in the Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (Serial Designation: D 86 – 25 T) of the American Society for Testing Materials. See p. 685.

[2] A shield conforming to these specifications is illustrated in the Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (Serial Designation: D 86 – 25 T) of the American Society for Testing Materials. See p. 685.

specified below. The flame should never be so large that it spreads over a circle of diameter greater than 4½ in. (11.43 cm.) on the under surface of the asbestos board. A sensitive regulating valve and gas pressure governor are desirable adjuncts, as they give complete control of heating.

Thermometer.

7. *Thermometer.*—The A.S.T.M. High Distillation Thermometer, conforming to the requirements specified in Section 6 (*b*) of the Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (Serial Designation: D 86 - 25 T) of the American Society for Testing Materials,[1] shall be used.

Graduates.

8. *Graduates.*—

(*a*) *For Sample.*—A graduate or pipette graduated to deliver 200 cc., ± 1.0 cc., may be used for measuring the sample to be tested.

(*b*) *For Fractions.*—The graduates used for measuring fractions shall be of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 cc. and the graduated portion shall be not less than 7 in. (17.78 cm.) nor more than 8 in. (20.32 cm.) in length. It shall be graduated in single cubic centimeters and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom at intervals of 10 cc. The overall height of the graduate shall be not less than 9¾ in. (24.8 cm.) nor more than 10¼ in. (26.0 cm.). The graduations shall not be in error by more than 1 cc. at any point on the scale.

PROCEDURE

Procedure.

9. (*a*) Provision shall be made for circulating water through the condenser.

(*b*) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft, lint-free cloth attached to a cord or copper wire may be used for this purpose.

(*c*) A volume of water-free oil equivalent to 200 cc. at 55 to 65° F. (12.8 to 18.3° C.) shall be placed in the flask.

(*d*) The thermometer, provided with a cork, shall be fitted tightly into the flask so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask.

(*e*) The charged flask shall be placed over the 2¾-in. (6.99-cm.) opening in the asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the vapor tube extends into the condenser tube not less than 1 in. (2.54 cm.) nor more than 2 in. (5.08 cm.).

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[1] See p. 685.

(*f*) A clean, dry graduated cylinder shall be placed at the outlet of the condenser tube in such position that the condenser tube shall extend into the graduate approximately 1 in. (2.54 cm.) but not below the 100-cc. mark.

Distillation.

10. When everything is in readiness, water shall be circulated through the condenser bath at such a rate that the water overflowing the condenser shall be at a temperature of 90 to 100° F. (32.2 to 37.8° C.). Heat shall be applied to the contents of the flask at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 10 nor more than 15 minutes. When the first drop falls from the end of the condenser, the reading of the distillation thermometer shall be recorded as the *initial boiling point.* The receiving cylinder shall then be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the distillation will proceed at a uniform rate of not less than 8 cc. nor more than 10 cc. per minute. A fraction shall be separated at every temperature point above the initial boiling point that is a multiple of 50° F.[1] When the temperature of the distilling vapors reaches 500° F. (260° C.), the temperature of the water in the condenser bath shall be adjusted to approximately 140° F. and the bath maintained between 140 and 160° F. (60 to 71.1° C.) for the remainder of the test. The distillation shall be continued until a vapor temperature of 700° F. (371.1° C.) is reached or until the maximum temperature point is reached. The maximum temperature point may result from complete distillation of the oil below 700° F. (371.1° C.), or from cracking.[2]

In case the oil distills completely below 700° F. (371.1° C.), the

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[1] When it is agreed to make allowance for the effect of barometric pressure, the observed temperature points at which fractions are to be separated in order to conform to multiples of 50° F. at 760 mm. shall be calculated by the Sydney Young equation, so expressed as to simplify the application:

$$C_f = 0.00012\ P(460 + t_f),$$

in which C_f is the correction to be added to the observed temperature when the barometer is above 760 mm. and to be subtracted from the observed temperature when the barometer is below 760 mm.

P is the difference in millimeters of mercury (at 32° F.) between 760 mm. and the observed barometric pressure, corrected for instrumental errors.

t is the temperature point to be corrected.

[2] Cracking will be evidenced by an increase in distilling rate with a thermometer reading that may advance very slowly, remain stationary, or recede, and an effort to adjust the distilling rate will usually result in a decided drop in the temperature reading. When this condition is observed, it is advisable to discontinue the distillation.

final adjustment of heat shall be made when a quantity of distillate amounting to 90 per cent of the sample has been collected. The heating shall be continued until the mercury reaches a maximum height and starts to fall consistently; but in no case should the period between the 90-per-cent point and the maximum temperature be more than 10 minutes. The highest temperature observed on the distillation thermometer shall be recorded as the *maximum temperature.*

Sufficient time shall be allowed after distillation is discontinued for the condenser to drain into the final fraction. Each fraction shall be brought to room temperature, the volume read and recorded, and the fraction set aside for the determination of the gravity. The percentage of distillate in each fraction shall be calculated and recorded. The difference between 100 and the sum of the distillates in per cent shall be calculated and recorded as *residue and loss.*

The gravity of each fraction and of the residue shall be determined at or corrected to 60° F. (15.6° C.). The gravity of each fraction shall be recorded as specific gravity or as gravity, degrees A. P. I.

SULFUR

Sulfur.

11. The sulfur shall be determined in accordance with the Tentative Method of Test for Sulfur in Petroleum Oils Heavier than Illuminating Oil (Serial Designation: D 129 - 22 T) of the American Society for Testing Materials.[1]

CARBON RESIDUE

Carbon Residue.

12. The carbon residue shall be determined in accordance with the Conradson Method, as described in the Tentative Method of Test for Carbon Residue of Lubricants (Serial Designation: D 189 - 24 T) of the American Society for Testing Materials.[2]

POUR POINT

Pour Point.

13. The pour point shall be determined in accordance with the Tentative Method of Test for Cloud and Pour Points of Petroleum Products (Serial Designation: D 97 - 25 T) of the American Society for Testing Materials.[3]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 777 (1922); also 1925 Book of A.S.T.M. Tentative Standards, p. 391.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol., 24, Part I, p. 899 (1924); also 1925 Book of A.S.T.M. Tentative Standards, p. 409.

[3] See p. 725.

VISCOSITY

Viscosity. 14. The viscosity shall be determined on the Saybolt Universal Viscosimeter at 100° F. in accordance with the Tentative Methods of Test for Viscosity of Petroleum Products and Lubricants (Serial Designation: D 88 – 25 T) of the American Society for Testing Materials.[1]

WATER

Water 15. Water shall be determined in accordance with the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95) of the American Society for Testing Materials.[2]

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[1] See p. 707

[2] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS
FOR
HIGH-CARBON TAR CEMENT.[1]

Serial Designation: D 110 – 25 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1923, 1925.

Scope. 1. These specifications cover material suitable for use in the construction of tar macadam and tar concrete pavements.

Properties. 2. The tar cement shall conform to the following requirements:[2]

(*a*) Water..0.00 per cent

(*b*) Softening point[3] (Ring-and-Ball Method)..30 to 40° C. (86 to 104° F.)[4]

(*c*) Distillation test:

Total Distillate, by weight, 0 to 170° C. (32 to 338° F.)	not more than 1.00	per cent
Total Distillate, by weight, 0 to 270° C. (32 to 518° F.)	not more than 10.00	"
Total Distillate, by weight, 0 to 300° C. (32 to 572° F.)	not more than 20.00	"
Residue, by weight	not less than 80.00	"

(*d*) Specific gravity at 25°/25° C. (77°/77° F.) of total distillate to 300° C. (572° F.)............................not less than 1.03

(*e*) Softening point (Ring-and-Ball Method) of residue from distillation test.......................not more than 65° C. (149° F.)

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] All tests shall be made on the sample as received, including the distillation test, and the results reported on a dry basis.

[3] If desired, a float test may be substituted for the softening point test, in which case the requirements shall be as follows: Float Test at 50° C. (122° F.), 100 to 220 sec.

[4] The specification range for softening point, within the above limits, should be 5° C. for any given locality, for example, 30 to 35° C. for cold climates, equivalent to a float test at 50° C. of 100 to 160 sec.; 35 to 40° C. for warm climates, equivalent to a float test at 50° C. of 160 to 220 sec.

(*f*) Total Bitumen (Soluble in Carbon Disulfide)[1]............ ....78 to 95 per cent

Methods of Testing.

3. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95);[2]

(*b*) *Distillation:* Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20;[2]

(*c*) *Softening Point:* Standard Method of Tests for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36);[2]

(*d*) *Float Test:* Tentative Method of Float Test for Bituminous Materials (Serial Designation: D 139 - 25 T);[3]

(*e*) *Total Bitumen:* Tentative Method of Test for the Determination of Bitumen (Serial Designation: D 4 - 23 T).[4]

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[1] The specification range for total bitumen covers a wide variety of materials. If products from vertical retort or low-carbon coke-oven tars are desired, a range of 88 to 95 per cent should be specified. If high-carbon coke-oven or mixtures of coke-oven and gas-house tars are desired, a range of 78 to 88 per cent should be specified.

[2] 1924 Book of A.S.T.M. Standards.

[3] See p. 748.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 751 (1923); also 1925 Book of A.S.T.M. Tentative Standards, p. 496.

Procedure when Determination of Suspension Clay is not Required.

5. In case it is not considered necessary to make a determination of the percentage of suspension clay, the following procedure for determination of total clay (clay plus suspension clay) may be followed:

An aliquot portion of the total volume of water used in washing the sample shall be taken and evaporated to dryness and the weighs of clay plus suspension clay obtained, which shall be expressed at the percentage of total clay in the original 25-g. sample.

TENTATIVE METHOD OF TEST FOR THE DETERMINATION OF MOISTURE EQUIVALENT OF SUBGRADE SOILS IN THE FIELD[1]

Serial Designation: D 220 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. This method covers the determination of the moisture equivalent of subgrade soils by means of a field test.

Apparatus. 2. The apparatus shall consist of a bowl, a spatula, and a suitable device for adding water dropwise.

Treatment of Sample. 3. A sample, weighing approximately 500 g., shall be taken from the field, air dried, and the lumps reduced in size to approximately $\frac{1}{8}$ in., all rock above this size being removed.

Procedure. 4. (*a*) The soil sample (prepared as described in Section 3) shall be placed in a bowl and water slowly added from a burette, the water and soil being mixed by means of a spatula until the soil reaches the consistency of putty and so the soil may be compacted with the spatula without any free water remaining on the surface. The soil shall then be struck off smooth and a drop of water allowed to fall on the surface. If this drop is immediately absorbed by the soil it indicates that the moisture equivalent has not been reached. A small increment of water shall then be added and thoroughly mixed with the soil. The surface shall again be struck off smooth and an additional drop allowed to fall thereon. This process is repeated until upon the addition of a drop of water the surface remains moist and has a shiny appearance.

By adding small increments of water and by watching the surface of the soil carefully, the critical point may readily be determined.

(*b*) The sample shall then be dried out at a temperature not exceeding 100° C. and the percentage of moisture determined on the basis of the dry weight of the sample.

NOTE.—This test is inaccurate when applied to soils whose moisture equivalent is below 20.

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

TENTATIVE METHOD OF FLOAT TEST
FOR
BITUMINOUS MATERIALS[1]

Serial Designation: D 139 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1923, 1924, 1925.

I. APPARATUS.

Float. 1. The float (Fig. 1) shall be made of aluminum or aluminum alloy and shall be in accordance with the following requirements:

	MINIMUM.	NORMAL.	MAXIMUM.
Weight of float, g	37.70	37.90	38.10
Total height of float, mm	34.0	35.0	36.0
Height of rim above lower side of shoulder, mm	26.5	27.0	27.5
Thickness of shoulder, mm	1.3	1.4	1.5
Diameter of opening, mm	11.0	11.1	11.2

Collar. 2. The collar (Fig. 1) shall be made of brass and shall be in accordance with the following requirements:

	MINIMUM.	NORMAL.	MAXIMUM.
Weight of collar, g	9.60	9.80	10.00
Over-all height of collar, mm	22.3	22.5	22.7
Inside diameter at bottom, mm	12.72	12.82	12.92
Inside diameter at top, mm	9.65	9.70	9.75

The top of the collar shall screw up tightly against the lower side of the shoulder.

Assembly. 3. The assembled float and collar, with the collar filled flush with the bottom and weighted to a total weight of 53.2 g., shall float upon water with the rim 8.5 ± 1.5 mm. above the surface of the water. Dimensions of the apparatus additional to those required above are given in Fig. 1.

Thermometer. 4. The thermometer shall conform to the following requirements. These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being 0 to 80° C. or 30 to 180° F., respectively.

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: -2 to $+80°$ C. in 0.2° C. or $+30$ to $+180°$ F. in 0.5° F.

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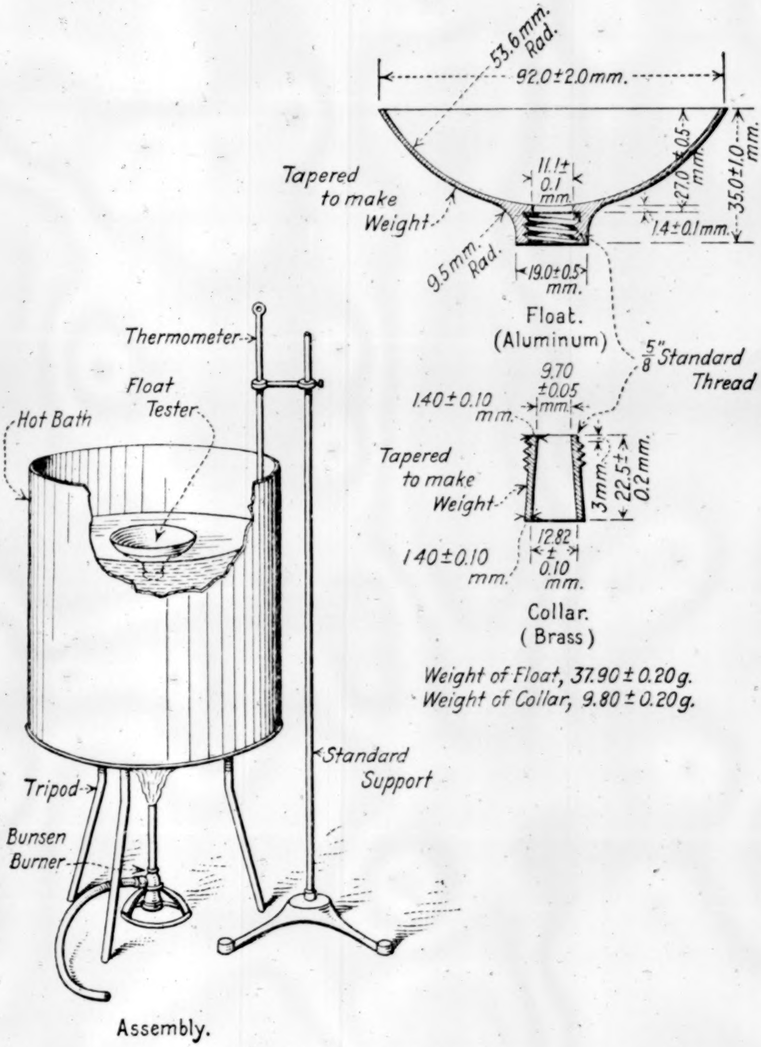
[1] Criticisms of this Tentative Method are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 9 to 14 mm. (0.35 to 0.55 in.).
Diameter, 4.5 to 5.5 mm. (0.18 to 0.22 in.).


53.6 mm. Rad.
92.0±2.0 mm.
Tapered to make Weight
11.1± 0.1 mm.
27.0±0.5 mm.
35.0±1.0 mm.
1.4±0.1 mm.
9.5 mm. Rad.
19.0±0.5 mm.
Float. (Aluminum)
5/8" Standard Thread
9.70 ±0.05 mm.
1.40±0.10 mm.
Tapered to make Weight
3 mm.
22.5± 0.2 mm.
12.82 ± 0.10 mm.
1.40±0.10 mm.
Collar. (Brass)
Thermometer
Float Tester
Hot Bath
Tripod
Bunsen Burner
Standard Support
Assembly.

Weight of Float, 37.90 ± 0.20 g.
Weight of Collar, 9.80 ± 0.20 g.

FIG. 1.—Float Test Apparatus.

DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 75 to 90 mm. (2.95 to 3.54 in.).

DISTANCE TO 80° C. OR 176° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).

TENTATIVE DEFINITIONS
OF
TERMS RELATING TO COAL[1]

Serial Designation: D 142 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1924, 1925.

Proximate Analysis.—In the case of coal, determination by prescribed methods of moisture, volatile matter, fixed carbon (by difference), and ash.

NOTE.—Unless otherwise specified, the term "proximate analysis" does not include determinations of sulfur or phosphorus or any determinations other than those named.

Ultimate Analysis.—In the case of coal, the quantitative determination of the elementary constituents carbon, hydrogen, and oxygen (the last by difference), in the material exclusive of incombustible residue; and the quantitative determination of sulfur and nitrogen in the material as a whole.

NOTE 1.—Determination of phosphorus is not by definition a part of ultimate analysis of coal, but may be specified when desired.

NOTE 2.—When the analysis is made on an undried sample, part of the hydrogen and oxygen as determined is present in the incidental or free moisture accompanying the coal. In comparing coals on the basis of their ultimate analysis, this fact is to be given due weight.

Moisture.—That constituent part, largely made up of water, in a substance, which is separated from it in analysis under specified conditions of drying. In the case of coal, the loss incurred by heating at 105° C. in accordance with the Standard Methods of Laboratory Sampling and Analysis of Coal (Serial Designation: D 22) of the American Society for Testing Materials.[2] As occurring in coal, moisture may or may not be apparent to the eye and touch.

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[1] Criticisms of these Tentative Definitions are solicited and should be directed to Mr. W. A. Selvig, Secretary of Committee D-5 on Coal and Coke, U. S. Bureau of Mines, 4800 Forbes St., Pittsburgh, Pa.

[2] 1924 Book of A.S.T.M. Standards.

Volatile Matter.—In the case of coal, that portion (exclusive of moisture) which is driven off by destructive distillation under the conditions specified in the Standard Methods of Laboratory Sampling and Analysis of Coal (Serial Designation: D 22) of the American Society for Testing Materials.[1] It is not contained in the coal as such, but is formed by decomposition, and consists of combustible matter (tars and gases) and incombustible matter whose respective proportions vary with the type of coal.

Fixed Carbon.—In the case of coal, the solid residue, other than ash, obtained by destructive distillation under the conditions specified in the Standard Methods of Laboratory Sampling and Analysis of Coal (Serial Designation: D 22) of the American Society for Testing Materials.[1] It is made up principally of carbon, but contains also appreciable quantities of sulfur, hydrogen, nitrogen and oxygen.

Ash.—Inorganic residue remaining after complete ignition of combustible substances. In the case of coal, the residue after igniting under the conditions specified in the Standard Methods of Laboratory Sampling and Analysis of Coal (Serial Designation: D 22) of the American Society for Testing Materials.[1]

NOTE.—The ash as thus determined is less in amount than that of the actual incombustible constituents of dry coal owing to chemical changes which occur in these by burning.

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[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE METHODS OF FLEXURE TESTING OF SLATE[1]

(DETERMINATION OF MODULUS OF RUPTURE AND MODULUS OF ELASTICITY)

Serial Designation: D 222 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Due to the unique properties of slate the flexure test is better adapted to use for strength and elasticity determinations than either compression or tension tests. Furthermore, several uses of slate are such that these determinations are of special interest and value, besides furnishing comparative data.

The property of slate termed "grain" causes a slab of the material to break transversely in one direction somewhat more readily than at right angles to this direction. For this reason it is desirable to test the strength and elasticity both parallel and perpendicular to the grain.

In the quarrying of slate, blasting is frequently resorted to and for this reason certain portions of the material may have been unduly strained. Low or erratic strength results on some of the test specimens should be regarded in the light of defective material.

A. Modulus of Rupture

I. TEST SPECIMENS

Number and Size of Specimens. 1. Six representative specimens, 12 by 1½ by 1 in. in size, of the particular slate under consideration shall be submitted for this test.

Preparation of Specimens. 2. The slate for test shall have been split to a thickness of approximately 1¼ in. and then sawed into strips 12 in. in length by 1½ in. in width. Half of these shall be cut with the length parallel to the grain and the other half with the length perpendicular to the grain. The 12 by 1½ in. faces shall then be planed to smooth surfaces and rubbed down to a thickness of approximately 1 in., care being taken to have the finished faces as nearly parallel as practical.

NOTE.—The shapes of specimens recommended for modulus of rupture and elasticity tests were determined partly by consideration of the proportions best adapted to the respective tests and partly on account of the convenience of preparing them from stock material. The small sizes recommended were first for economy of material and second, to permit the use of lighter testing equipment. Where only the large machines are available it is desirable, in order to obtain consistent results, to use larger specimens. In such cases approximately the same proportionate dimensions should be adhered to for the respective tests.

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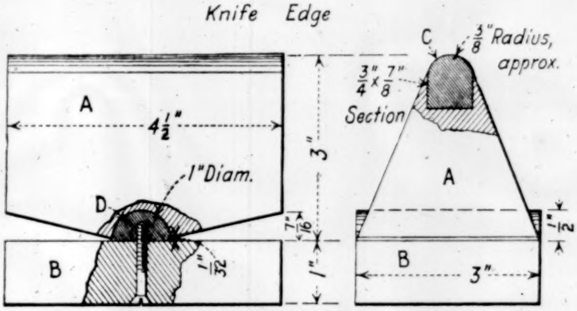
[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. D. K. Boyd, Secretary of Committee D-16 on Slate, Structural Service Bureau, Otis Building, Philadelphia.

II. PROCEDURE

Drying. 3. Before testing, the specimens shall be dried in an oven at a temperature of 110 to 120° C. for 24 hours.

Measuring Specimens. 4. The thickness and the width of the specimen at the middle shall be measured to the nearest 0.01 in.

Testing. 5. The testing machine shall be sensitive to 5 lb. when loaded to 500 lb. The specimen shall be supported flatwise on knife edges of the type shown in Fig. 1, spaced 10 in. on centers. The load shall be applied by a third knife edge at the middle of the span. The rate of loading shall be approximately 100 lb. per minute. The breaking load shall be recorded to the nearest 5 lb.



A, B = Cast Iron Wedges.
C, D = Cold-Rolled Steel.

FIG. 1.

Computation of Results. 6. The modulus of rupture shall be calculated from the formula:

$$M_f = \frac{3}{2}\frac{wl}{bd^2}$$

where w = breaking load in pounds, l = length of span in inches, b = width of specimen in inches, and d = thickness of specimen in inches.

Reporting Results. 7. The average value of the three specimens cut parallel to the grain shall be reported as the modulus of rupture parallel to the grain, and the average value of the other three, as the modulus of rupture perpendicular to the grain. In case any specimen shows a value 25 per cent lower than the average of its group, it shall be examined for defects and if the low value appears to be due to an imperfection, the average of the other two shall be reported as the modulus of rupture of the group. All determinations shall be reported as information.

NOTE.—The following method of marking the specimens before testing is recommended:

By means of a tri-square the middle section and two end sections, 5 in. distant from the center, may be circumscribed. These marks serve as a guide to the operator in properly centering and aligning the specimen in the testing machine.

to 30 Penetration, for Use in Sheet Asphalt and Asphaltic-Concrete Pavements (Serial Designation: D 163 – 23 T) of the American Society for Testing Materials.[1]

(*c*) The matter insoluble in pure benzol shall be granular mineral matter which after recovery from the mastic cake shall conform to the following requirements as to grading.

Passing 200-mesh sieve	not less than 25 per cent
Passing 10-mesh sieve, retained on 50-mesh sieve	not more than 25 "
Retained on 10-mesh sieve	not more than 1 "

It shall contain not more than 5 per cent by weight of material soluble in hydrochloric acid.

Asphalt Cement. 4. The asphalt cement shall conform to the requirements of the Tentative Specifications for Asphalt Cement, 25 to 30 Penetration, for Use in Sheet Asphalt and Asphaltic-Concrete Pavements (Serial Designation: D 163 – 23 T) of the American Society for Testing Materials.[1]

Mineral Aggregate. 5. The mineral aggregate shall be gravel or crushed stone and sand and free from carbonates. It shall conform to the following requirements as to grading:

Passing 200-mesh sieve	none
Passing 50-mesh sieve, retained on 200-mesh sieve	nor more than 25 per cent
Passing 10-mesh sieve, retained on 50-mesh sieve	not less than 25 "
Passing 4-mesh sieve, retained on 10-mesh sieve	not less than 50 "
Retained on 4-mesh sieve	not more than 10 "

It shall contain not more than 5 per cent by weight of material soluble in hydrochloric acid.

Method of Testing. 6. The properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (Serial Designation: D 147 – 25 T) of the American Society for Testing Materials.[2]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 721 (1923); also 1925 Book of A.S.T.M. Tentative Standards, p. 454.

[2] See p. 786.

TENTATIVE SPECIFICATIONS
FOR
SMOOTH-SURFACED ASPHALT ROLL-ROOFING[1]

Serial Designation: D 224 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. These specifications cover smooth-surfaced roll-roofing (medium and heavy weights), either 36 in. or 32 in. in width, composed of roofing felt saturated and coated on both sides with asphalt, and surfaced top and bottom with powdered talc or mica. The medium weight roofing is intended for use on semi-permanent structures and the heavy weight roofing for use on permanent structures.

I. MANUFACTURE

Manufacture. 2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with an asphaltic saturant, then coated on both sides with an asphaltic coating, and finally surfaced on both sides with powdered talc or mica.

Character of Felt. 3. The felt shall be a roofing felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth, and upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

Character of Saturant and Coatings. 4. The saturant and coatings shall be composed principally of asphaltic materials, and the presence of saponifiable constituents shall be permitted.

Character of Felt after Saturation. 5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Character of Mineral Surfacings. 6. The surfacing shall be non-combustible material such as powdered talc or mica.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

roofing not less than 1 pint. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased by $\frac{1}{4}$ pint per "square."

V. SAMPLING AND TESTING

14. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles (Serial Designation: D 228 – 25 T) of the American Society for Testing Materials.[1]

VI. INSPECTION

15. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

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[1] See p. 792.

TENTATIVE SPECIFICATIONS
FOR
SLATE-SURFACED ASPHALT ROLL-ROOFING AND SLATE-SURFACED ASPHALT SHINGLES[1]

Serial Designation: D 225 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope.

1. These specifications cover slate-surfaced roll roofing, either 36 or 32 in. in width, and slate-surfaced composition shingles, composed of roofing-felt saturated and coated on both sides with asphalt and surfaced on the weather side with granulated slate (green, red, black, or such color or combinations of color as may be agreed upon between the purchaser and seller), or similar mineral material to be specified by the purchaser, and on the reverse side with powdered talc or mica to prevent the roofing or shingles adhering together in the package. Such roofing and shingles are intended for use on semi-permanent and permanent buildings.

I. MANUFACTURE

Manufacture.

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with an asphaltic saturant, then coated on both sides with an asphaltic coating, and finally surfaced on the weather side with granulated slate or similar mineral material to be specified by the purchaser, and on the reverse side with powdered talc or mica.

Character of Felt.

3. The felt shall be a roofing felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth, and upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

IV. PACKING

13. (*a*) Slate roll-roofing shall be put up in rolls of 108 sq. ft. No rolls shall contain more than two pieces nor shall there be more than 3 per cent of rolls containing two pieces in any shipment. The rolls shall be tightly wound and securely wrapped in a substantial grade of paper. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the roll shall be protected with metal caps, cloth or other suitable means. The nails and lap-cement shall be furnished either in each roll or in bulk, as specified in the order. When furnished in bulk, the cloth or metal ends may be omitted. Rolls for export shipment shall be packed as agreed upon by the purchaser and seller.

Shingles shall be packed in bundles weighing not more than 135 lb. gross. The bundles shall either be packed in cardboard cartons or between wooden or cardboard ends and tied with rope, wire or steel bands so as to make a substantial package, as may be agreed upon by the purchaser and seller. Each roll or package shall be plainly marked with the manufacturer's name and brand or as agreed upon by the purchaser and seller, and shall contain printed directions for laying.

(*b*) *Nails.*—The gage of the wire shall not be greater than "A. S. & W." No. 10 (0.135 in.) nor thinner than No. 12 (0.105 in.); the head shall be not less than $\frac{3}{8}$ in. in diameter, and not less than 0.025 in. thick; the shank shall be between $\frac{3}{4}$ and 1 in. long for slate-roll roofing, and for shingles 1 in. long when laid on solid sheathing, or $1\frac{1}{2}$ to $1\frac{3}{4}$ in. when laid over old shingles. The lower end of the shank shall be pointed, and either smooth or barbed, and if the latter, then the barbings shall not increase the diameter of the lower two-thirds more than 0.01 in. The nails shall be zinc coated with not less than $\frac{1}{2}$ oz. nor more than $1\frac{1}{4}$ oz. per sq. ft. of nail surface. Hot-galvanized, electrogalvanized, or sherardized coatings will be accepted. There shall be furnished per "square" of slate-roll roofing not less than an average of 252 nails for 36 in., and not less than an average of 275 nails for 32-in. roofing. For shingles, the nails shall be purchased separately, and the number required per "square" will depend upon the size and form of the shingles and shall be agreed upon by the purchaser and seller.

(*c*) *Lap-cement* (*for Slate Roll.*)—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect upon the roofing. The cement shall have a minimum flash point of

71° F. (21.5° C.) when tested by the Tagliabue Closed-cup Tester. There shall be furnished for each square of 36-in. roofing not less $\frac{3}{4}$ pint and for 32-in. roofing not less than 1 pint. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased $\frac{1}{4}$ pint per "square".

V. SAMPLING AND TESTING

14. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles (Serial Designation: D 228 - 25 T) of the American Society for Testing Materials.[1]

VI. INSPECTION

15. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

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[1] See p. 792.

TENTATIVE SPECIFICATIONS
FOR
ASPHALT-SATURATED ROOFING FELT FOR USE IN WATERPROOFING AND IN CONSTRUCTING BUILT-UP ROOFS[1]

Serial Designation: D 226 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope.

1. These specifications cover bituminized felted fabrics, either 36 or 32 in. in width, composed of roofing felt saturated, but not coated, with asphalt for use in the membrane system of waterproofing and in the construction of built-up roofs.

I. MANUFACTURE

Manufacture.

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with an asphaltic saturant.

Character of Felt.

3. The felt shall be a roofing-felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth, and upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

Character of Saturant.

4. The saturant shall be composed of asphaltic materials.

Character of Felt after Saturation.

5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2 in. strips at random across the entire sheet and splitting them open for their full length.

Surfacing.

6. The surface of the fabric shall not be coated or covered with talc or other substance which would tend to interfere with the adhesion between the fabric and the plying cement.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

II. PHYSICAL PROPERTIES

Fabricated Bituminized Felt.

7. The fabricated bituminized felt shall conform to the following requirements:

(*a*) *Width of Roll.*—Either 36 or 32 in., as may be specified, ± 1/4 in.

(*b*) *Gross Weight per Roll.*—40 to 80 lb.

(*c*) *Weight of Wrapping and Packing Material per Roll.*—Maximum 1/2 lb.

(*d*) *Weight of Material per 100 sq. ft. exclusive of Packing and Wrapping.*—14 lb. ± 1 lb.

(*e*) *Moisture.*—Maximum, 2.5 per cent of the net weight.

(*f*) *Loss on Heating (Exclusive of Moisture) at 105° C. (221° F.) for 5 hours.*—Maximum, 2 per cent.

(*g*) *Pliability at 77° F. (25° C.).*—Shall not crack when bent flat on itself.

(*h*) *Saturant in Moisture-Free Felt.*—Minimum 140 per cent by weight.

Desaturated Felt.

8. The desaturated felt shall conform to the following requirements:

(*a*) *Weight of Moisture-Free Desaturated Felt.*—The average "Number" (expressed on the basis of pounds per 480 sq. ft.) shall be 27, equivalent to 5.625 lb. per 100 sq. ft., with a permissible variation of 10 per cent.

(*b*) *Average Thickness of Desaturated Felt.*—Minimum, 0.025 in.

(*c*) *Ash in Moisture-Free Desaturated Felt.*—Maximum, 10 per cent.

III. WORKMANSHIP AND FINISH

Freedom from Defects

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations.

Stickiness.

10. The rolls shall not crack, or be sticky enough to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50° F. (10° C.).

IV. PACKING

11. The rolls of saturated felt need not be wound on cores, but they shall be securely wrapped in a substantial grade of paper of the same width as the fabric. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent

it shifting from position. The ends of the roll need not be covered. As an alternative, the rolls of saturated felt may either be tied with strings or simply pasted at the ends, as may be agreed upon by the purchaser and seller. No roll shall contain more than two pieces, and there shall not be more than 3 per cent of rolls containing two pieces in any shipment. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed by the purchaser and seller, and shall bear a notation showing the type of saturant.

V. SAMPLING AND TESTING

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (Serial Designation: D 146 - 25 T) of the American Society for Testing Materials.[1]

VI. INSPECTION

13. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

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[1] See p. 776.

TENTATIVE SPECIFICATIONS
FOR
COAL-TAR SATURATED ROOFING FELT FOR USE IN WATERPROOFING AND IN CONSTRUCTING BUILT-UP ROOFS[1]

Serial Designation: D 227 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. These specifications cover bituminized felted fabrics, either 36 or 32 in. in width, composed of roofing felt saturated, but not coated, with refined coal tar, for use in the membrane system of waterproofing and in the construction of built-up roofs.

I. MANUFACTURE

Manufacture. 2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a coal-tar saturant.

Character of Felt. 3. The felt shall be a roofing-felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth, and upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

Character of Saturant. 4. The saturant shall be composed of coal tar from which the highly volatile constituents have been removed.

Character of Felt after Saturation. 5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Surfacing. 6. The surface of the fabric shall not be coated or covered with talc or other substance which would tend to interfere with the adhesion between the fabric and the plying cement.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

II. PHYSICAL PROPERTIES

Fabricated Bituminized Felt.

7. The fabricated bituminized felt shall conform to the following requirements:

(*a*) *Width of Roll.*—Either 36 or 32-in., as may be specified, ± ¼ in.

(*b*) *Gross Weight per Roll.*—40 to 80 lb.

(*c*) *Weight of Wrapping and Packing Material per Roll.*—Maximum, ½ lb.

(*d*) *Weight of Material per 100 sq. ft. exclusive of Packing and Wrapping.*—14 lb. ± 1 lb.

(*e*) *Moisture.*—Maximum, 2.5 per cent of the net weight.

(*f*) *Distillate*, per cent by weight to 210° C., calculated on extracted saturant, not more than 2.

Distillate, per cent by weight to 235° C., calculated on extracted saturant, not more than 5.

(*g*) *Pliability at 77° F. (25° C.).*—Shall not crack when bent flat on itself.

(*h*) *Saturant in Moisture-Free Felt.*—Minimum, 140 per cent by weight.

Desaturated Felt.

8. The desaturated felt shall conform to the following requirements:

(*a*) *Weight of Moisture-Free Desaturated Felt.*—The average "Number" (expressed on the basis of pounds per 480 sq. ft.) shall be 27, equivalent to 5.625 lb. per 100 sq. ft., with a permissible variation of 10 per cent.

(*b*) *Average Thickness of Desaturated Felt.*—Minimum, 0.025 in.

(*c*) *Ash in Moisture-Free Desaturated Felt.*—Maximum, 10 per cent.

III. WORKMANSHIP AND FINISH

Freedom from Defects.

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations.

Stickiness.

10. The rolls shall not crack, or be sticky enough to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50° F. (10° C.).

IV. PACKING

11. The rolls of saturated felt need not be wound on cores, but they shall be securely wrapped in a substantial grade of paper of the same width as the fabric. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the roll need not be covered.

As an alternative, the rolls of saturated felt may either be tied with strings or simply pasted at the ends, as may be agreed upon by the purchaser and seller. No roll shall contain more than two pieces, and there shall not be more than 3 per cent of rolls containing two pieces in any shipment. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed by the purchaser and seller, and shall bear a notation showing the type of saturant.

V. SAMPLING AND TESTING

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (Serial Designation: D 146 – 25 T) of the American Society for Testing Materials.[1]

VI. INSPECTION

13. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

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[1] See p. 776.

TENTATIVE METHODS OF TESTING FELTED AND WOVEN FABRICS SATURATED WITH BITUMINOUS SUBSTANCES FOR USE IN WATERPROOFING AND ROOFING[1]

Serial Designation: D 146 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1922; Revised, 1923, 1925.

Scope. 1. These methods cover the examination of felted or woven fabrics saturated (but not coated) with asphalt or coal-tar products, for use in the "membrane" system of waterproofing.

I. SAMPLING.

Sampling. 2. From each shipment or portion thereof representing a product of the same kind, class and weight, a number of rolls shall be selected at random, equivalent to the cube root of the total number of rolls in the lot. If the cube root, as calculated, proves to be a fractional number, it shall be expressed as the next higher whole number. For convenience, the following table is given, showing the number of samples to be selected for shipments of various sizes:

Packages in Shipment.	Number of Packages Selected.	Packages in Shipment.	Number of Packages Selected.
2 to 8	2	1332 to 1728	12
9 to 27	3	1729 to 2197	13
28 to 64	4	2198 to 2744	14
65 to 125	5	2745 to 3375	15
126 to 216	6	3376 to 4096	16
217 to 343	7	4097 to 4913	17
344 to 512	8	4914 to 5832	18
513 to 729	9	5833 to 6859	19
730 to 1000	10	6860 to 8000	20
1001 to 1331	11	8001 to 9261	21

II. EXAMINATION OF FINISHED FABRIC.

Width. 3. The width of each roll taken shall be measured to the nearest $\frac{1}{32}$ in. The minimum, maximum and average width so obtained shall be reported.

Gross Weight per Roll. 4. Each roll taken shall be weighed intact to the nearest $\frac{1}{4}$ lb. and the minimum, maximum and average gross weight shall be reported.

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[1] Criticisms of these Tentative Methods are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

Wrapping and Packing Materials.

5. Each roll sampled shall be stripped. All the wrappers and packing material shall be weighed together to the nearest $\frac{1}{4}$ lb. and the average weight shall be reported. Each roll should be temporarily fastened with a piece of light string and handled carefully, so as not to disturb any detached surfacing material.

Net Weight per Roll.

6. The minimum, maximum and average net weight per roll shall be calculated by subtracting the average weight of the wrapping and packing material as determined in Section 5 from the average gross weight as determined in Section 4.

Weight per Unit Area.

7. Each roll sampled shall be laid flat, the first convolution or two carefully unwound, and with a knife and straight edge the sheet shall be cleanly cut across at right angles to the edges. A section measuring 30 in. in the direction of the roll's length shall be removed. If the material is surfaced with sand or other fine material any detached particles belonging to each 30-in. section should be retained. The width of each section shall be measured to the nearest $\frac{1}{32}$ in. Each section, together with any detached surfacing, shall be weighed to the nearest gram. The weight in pounds per 100 sq. ft. or the weight in ounces per sq. yd. shall be calculated from the formulas:

$$\text{Pounds per 100 sq. ft.} = 1.0582 \times \frac{\text{Weight of 30-in. section in grams}}{\text{Width of 30-in. section in inches}}$$

$$\text{Ounces per sq. yd.} = 1.5238 \times \frac{\text{Weight of 30-in. section in grams}}{\text{Width of 30-in. section in inches}}$$

From the results obtained for each roll sampled, the minimum, maximum, and average weight shall be calculated.

NOTE.—As a referee method or in case any dispute arises regarding the weight "per unit area," the method described in Section 8 shall be followed. From the net weight of each roll determined as in Section 6, the weight per unit area may be calculated as a check.

Area per Roll.

8. The average area per roll in square feet shall be calculated by dividing the average net weight per roll, as determined in Section 6, by the average weight in pounds per 100 sq. ft. as determined in Section 7, and multiplying by 100.

NOTE.—As a referee method or in case any dispute arises regarding the "area per roll" as calculated, each roll sampled shall be unwound and the total length measured within $\frac{1}{4}$ in. with a steel tape. From the width of each roll, as determined in Section 3, the "area per roll" may be accurately calculated as a check and the minimum, maximum and average for the shipment calculated.

Detached Comminuted Surfacing.

9. If the material is surfaced with sand or other finely comminuted material, the surfacing shall be swept with a moderately stiff brush from all the 30-in. sections taken. All the material thus removed shall be caught and weighed to the nearest ounce. From the aggregate areas of all the sections taken, the average weight of detached comminuted surfacing shall be calculated in pounds per 100 sq. ft.

Thickness. 10. The thickness of each specimen shall be measured at 10 equally spaced points, 1 in. from a cut edge, with a micrometer gage having flat bearing surfaces at both contact points of not less than ½ in. in diameter and preferably of the spring-and-dial type. The readings for each roll sampled shall be averaged and the minimum, maximum and average thickness calculated to the nearest 0.001 in.

Moisture. 11. From each specimen, 2-in. strips shall be cut as shown at *A-1* and *A-2* in Fig. 1. The 2-in. strips shall be cut into 1-in. squares. About 50 g., selected at random, shall be accurately weighed and distilled with 100 cc. of a coal-tar distillate of which 5 to 10 per cent boils below 100° C. (212° F.) and of which at least 90 per cent distills up to 180° C. (356° F.) in an apparatus of the form and under the conditions prescribed in the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95) of the American Society for Testing Materials.[1] The flask containing the solvent and sample shall be heated in a paraffin bath at a temperature of 302 to 338° F. (150 to 170° C.) until no further water passes over. The distillate shall be allowed to stand in the graduated receiver until the water separates, whereupon the volume of water shall be measured and its weight calculated. From this, the

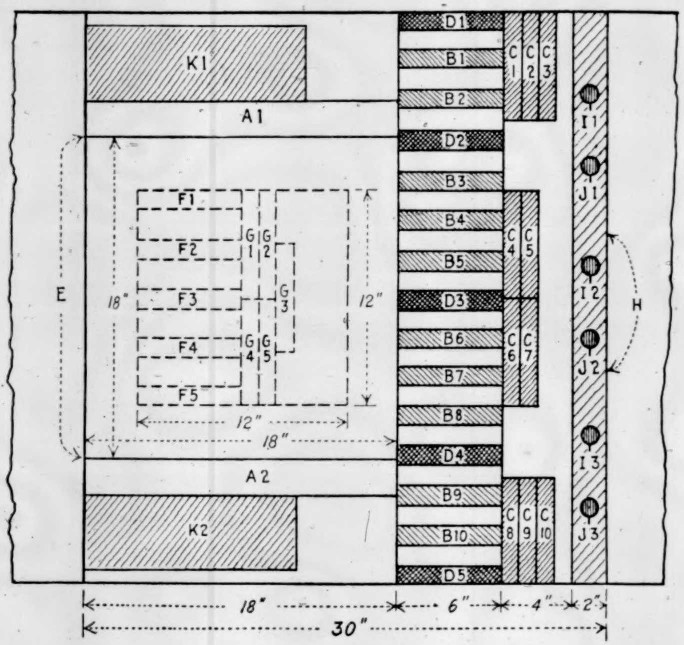

FIG. 1

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[1] 1924 Book of A.S.T.M. Standards.

average percentage of moisture in the fabric, as received, shall be calculated.

Strength.

12. (*a*) *Felted Fabrics.*—With a photographic trimmer having a stop attached exactly 1 in. behind the blade, ten strips shall be cut with the fiber grain, as shown at *B-1* to *B-10* (Fig. 1) and ten strips across the fiber grain as shown at *C-1* to *C-10*. Each strip should measure 6 in. and have parallel sides 1 in. apart (within $\frac{1}{32}$ in.) with the edges cut straight and clean. Both sets of strips shall be tested at 70° F. (21.1° C.), using a tension testing machine such as the Scott Strength Tester (Type F, 100 lb. capacity), the Perkins Strip Tester, or equal, in which the clamps are attached to swivels free to move in any direction. The test strips shall be gripped $1\frac{1}{2}$ in. on each end, leaving approximately 3 in. between the clamps. The tension shall be increased at a uniform speed of 3 lb. per second, using a metronome. If any strip breaks nearer than $\frac{1}{2}$ in. to either clamp, the reading shall be disregarded, and an additional strip shall be tested in its place. The ten readings, with and across the grain, respectively, shall be averaged for each roll sampled. From these results the average strength with and across the fiber grain, respectively, of the fabric as supplied, shall be calculated.

(*b*) *Woven Fabrics*—Woven fabrics shall be tested at 70° F. (21.1° C.) in accordance with the "grab test No. 2" as described in Section 12 (*b*) of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[1]

NOTE.—As a referee method, or in case any dispute arises regarding the strength, the test shall be repeated, with the exception that the fabric before testing shall be exposed at least two hours in an atmosphere of 65 per cent relative humidity at 70° F. (21.1° C.).

Pliability.

13. With the trimmer described in Section 12 (*a*), five 6-in. strips shall be cut with the fiber grain, as shown at *D-1* to *D-5* (Fig. 1) and immersed in water at 77° F. (25° C.) for from 10 to 15 minutes. These strips shall be bent through 180 deg. at a uniform speed, in exactly 2 seconds, around a mandril, the diameter of which shall be as follows: The first, around a mandril 25 mm. in diameter; the second, around a mandril 20 mm. in diameter; the third, around a mandril 15 mm. in diameter; the fourth, around a mandril 10 mm. in diameter, and the fifth, around a mandril 5 mm. in diameter. The test may also be made at 32° F. (0° C.). The pliability shall be expressed numerically from 1 to 10 as follows:

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[1] 1924 Book of A.S.T.M. Standards.

(1) Cracks entirely through the sheet on the 25-mm. mandril.
(2) Cracks part way through the sheet on the 25-mm. mandril.
(3) Cracks on the 25-mm. mandril.
(4) Cracks on the 20-mm. mandril.
(5) Cracks on the 15-mm. mandril.
(6) Cracks on the 10-mm. mandril.
(7) Cracks on the 5-mm. mandril.
(8) Cracks when bent through 180 deg. flat on itself.
(9) May be bent through 180 deg. flat on itself in one direction without cracking, but will crack when bent through 360 deg. in the opposite direction.
(10) May be bent through 180 deg. flat on itself in one direction and then through 360 deg. flat on itself in the opposite direction without cracking.

The readings for each roll shall be averaged separately, and from these results the minimum, maximum and average pliability of the felt shall be calculated.

Water Absorbed.

14. (*a*) A section 18 in. square (as shown at *E*, Fig. 1) shall be cut from each specimen, weighed, and completely immersed in distilled water at 77° F. (25° C.) for 24 hours. The specimen shall then be removed and dried superficially by pressing lightly between two towels. As the moisture enters through the edges of the sheet more rapidly than through the surfaces, each specimen shall be trimmed to exactly 12 in. square, representing four-ninths of the original area, and reweighed rapidly. The increase in weight shall be calculated on the basis of the original test specimen by multiplying by $2\frac{1}{4}$. The percentage increase in weight represents the water absorption. The minimum, maximum and average for the shipment shall be calculated.

(*b*) Five strips shall be cut with the fiber grain as shown at *F 1* to *F 5* (Fig. 1); five other strips shall be cut across the fiber grain as shown at *G 1* to *G 5* (Fig. 1) and the strength shall be redetermined as in Section 12. The decrease in strength in percentage shall be calculated after the specimen has been subjected to water.

Loss on Heating.

15. (*a*) From each specimen, 12 by 6-in. strips shall be cut at *K-1* and *K-2* in Fig. 1, care bring taken not to disturb any of the detached surfacing. Each strip shall be weighed and suspended in the center of an air oven maintained at 221° F. (105° C.) ± 5° F. (3° C.) with a thin wire fastened through holes punctured near one edge. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The strips shall be kept in the oven for exactly 5 hours, then cooled and removed carefully, and each specimen weighed. The average loss shall be

calculated as a percentage and the average percentage of moisture (ascertained in Section 11) deducted. The final figure shall represent the average loss on heating, exclusive of moisture.

Extraction of Saturant.

(*b*) *Coal-Tar Saturated Felt Only.*—From the rolls making up the sample, strips 6 in. wide shall be cut across the sheet. Sufficient of these shall be taken to make up a sample of 250 g. ±5.0 g. These strips then shall be rolled up and placed in the wire basket of the extraction apparatus described in the Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (Serial Designation: D 147 - 25 T) of the American Society for Testing Materials[1] or other extraction apparatus of suitable construction. The sample shall then be covered with a disk of soft filter paper to insure an even distribution of the solvent. The extraction shall be carried out as described in Methods D 147 - 25 T.

Recovery of Saturant from CS_2 Extract.

(*c*) The CS_2 extract containing the bitumen shall be transferred to a 500-cc., short-neck, round-bottom flask. The flask shall be equipped with a Hempel column, 16 cm. in length, 15 mm. in inside diameter and filled to a depth of 5 cm. with glass beads. The Hempel column shall be connected with a water cooled condenser. A 0 - 80° C. thermometer as described in the Tentative Method of Float Test for Bituminous Materials (Serial Designation: D 139 - 25 T) of the American Society for Testing Materials[2] shall be placed in the Hempel column so that the top of the bulb shall be opposite the bottom of the tubulature. Distillation for the removal of CS_2 shall be continued by means of a steam bath until drops cease to fall from the condenser. The distillation then shall be carried out over a small flame, heating carefully and observing the thermometer until the distillation ceases and the temperature of the vapor as indicated by the thermometer shows no further rise on slightly increasing the flame.

Distillation of Saturant.

(*d*) When the contents of the flask is sufficiently cool to be poured without appreciable vaporizing, 100 g. ±0.1 g. shall be distilled in accordance with the method described in Sections 10, 11 and 12 of the Standard Methods of Sampling and Analysis of Creosote Oil (Serial Designation: D 38) of the American Society for Testing Materials.[3]

Fractions shall be taken at 210° C. and 235° C. and the distillation stopped at that point. The per cent of distillate by weight at each temperature shall be calculated on the basis of the weight of sample taken for distillation.

III. DESATURATING THE FABRIC.

Desaturated Fabric.

16. From each roll sampled, a 2-in. strip (within $\frac{1}{32}$ in.) shall be cut across the specimen as shown at *H* (Fig. 1). Each strip shall be extracted with c. p. benzol in an extractor described in Section 4 of

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[1] See p. 786. [2] See p. 748. [3] 1924 Book of A.S.T.M. Standards.

the Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (Serial Designation: D 147 - 24 T) of the American Society for Testing Materials,[1] the extraction being continued for several hours after the drippings have become colorless. The desaturated fabric shall be removed and heated in a ventilated oven at 225° F. (107.1° C.), cooled in a desiccator, any adhering comminuted surfacing being brushed off and retained, and weighed as rapidly as possible. The heating shall be repeated until the weight of the fabric remains constant as determined by two consecutive weighings taken not less than ten minutes apart which shall show a further loss of not more than 0.1 per cent. This will give the weight of the fabric in the moisture-free state. Where a coal-tar pitch saturant has been used, the moisture-free weight of the desaturated fabric shall be corrected for the carbonaceous matter retained mechanically in its interstices, by means of the method described in Section 17.

Retained Carbonaceous Matter.

17. The following colorimetric method shall be used to ascertain the carbonaceous matter derived from a coal-tar pitch saturant and retained by the desaturated fabric:

(*a*) About 15 g. of an unsaturated fabric of the same general character as the one under examination, shall be macerated by boiling in water, disintegrating with a rotary egg-beater and picking the fibers apart with needles. The fibers shall be filtered through fine cloth and dried at a temperature of 225° F. (107.1° C.) to constant weight. One gram of the fibers shall be accurately weighed into a flask and diluted to exactly 100 cc. with distilled water at room temperature. About 50 g. of glass beads shall be added and the contents of the flask shaken vigorously until the fibers are reduced to a homogeneous pulp in uniform suspension.

(*b*) A distilled coal-tar, having approximately 10 to 25 per cent of insoluble carbonaceous matter, shall be procured and the carbonaceous matter extracted with benzol until it is free from soluble matter; then dried at a temperature of 225° F. (107.1° C.) to constant weight. One gram of the purified carbonaceous matter shall be accurately weighed and diluted to exactly 100 cc. at room temperature with a starch solution of a consistency sufficient to carry the carbonaceous matter into temporary suspension. (A 12.5-per-cent solution is recommended.)

(*c*) The liquid carrying the fibers, obtained as described in Paragraph (*a*), shall be titrated with the suspension of carbonaceous matter, obtained as described in Paragraph (*b*), and from time to time a field prepared from a drop of the well agitated mixture shall be examined under a microscope at 100 diameters magnification until the color exactly matches a field prepared from the desaturated fabric under examination (obtained as described in Section 16), when both are viewed side by side under parallel conditions. The end-point is fairly sharply defined. The burette reading gives directly the percentage of carbonaceous matter adhering to the moisture-free fabric under examination.

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[1] See p. 786.

(*d*) The weight of moisture-free fabric ascertained in Section 16 shall be corrected by deducting the weight of adhering carbonaceous matter.

Total Comminuted Surfacing.

18. The total comminuted surfacing plus any filler present in the bituminous saturant represents the sum of the detached comminuted surfacing (Section 9), the amount brushed off from the surface of the desaturated fabric (Section 16), plus the amount recovered upon evaporating and igniting the bituminous extract obtained in accordance with Section 16.

Bituminous Saturant.

19. The weight of bituminous saturant represents the difference between the weight of the original fabric (Section 7), and the sum of the weights of the moisture-free desaturated fabric (Sections 16 and 17), plus moisture (Section 11), plus the total comminuted surfacing and filler present (Section 18).

IV. EXAMINATION OF THE EXTRACTED FABRIC.

(*A*) *Woven Fabrics.*

Weight.

20. Woven fabrics shall be tested for weight and number of threads per in. in accordance with Sections 6 to 11, inclusive, of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[1]

(*B*) *Felted Fabrics.*

Weight.

21. The weight in pounds per 480 sq. ft. of the desaturated felt (corrected for any carbonaceous matter present) for each specimen examined, shall be calculated separately from the formulas:

$$\text{“Number”} = \frac{\text{Weight of specimen in grams}}{\text{Area of specimen in square centimeters}} \times 983$$

or

$$\text{“Number”} = \frac{\text{Weight of specimen in grams}}{\text{Area of specimen in square inches}} \times 152$$

This represents the "number" of the moisture-free felt on the felt makers' scale. The minimum, maximum and average "number" of the felt in its moisture-free state shall be calculated. This may be corrected to the condition in which it actually existed in the material under examination, by adding thereto the percentage of moisture as determined in Section 11.

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[1] 1924 Book of A.S.T.M. Standards.

Ash. 22. A representative sample shall be secured by cutting from each strip of desaturated felt a piece about ½ in. in diameter as shown at *I-1*, *I-2*, and *I-3* (Fig. 1). About 25 g. selected at random from all the specimens sampled in this manner shall be accurately weighed, and incinerated in a weighed porcelain or quartz crucible either over an open flame or in a muffle, until all the carbon is consumed. A few drops of ammonium carbonate solution shall be added, the sample ignited gently, and weighed. The percentage of ash shall be calculated on the basis of the moisture-free felt.

NOTE.—As a referee method, or in case greater accuracy is desired, the three portions taken from each specimen roll shall be weighed and ignited separately. The minimum, maximum and average of ash present shall be calculated on the basis of the moisture-free felt.

Fibers Present. 23. (*a*) A representative sample shall be secured by cutting from each strip of desaturated felt a piece about ½ in. in diameter as shown at *J-1*, *J-2*, and *J-3* (Fig. 1). The pieces shall be placed in a small beaker, boiled with water and macerated until the fibers are well separated and reduced to a pulp. They shall then be transferred to a stout flask. About 50 g. of glass beads shall be added and the contents of the flask shaken vigorously until the fibers are reduced to a homogeneous mass in a uniform suspension. A small portion of the well-mixed pulp shall be removed with a dropper or pipette and three or four drops shall be rapidly transferred to a microscope slide. Ten such slides shall be prepared, refilling the dropper each time.

(*b*) The drops shall be spread thinly over the microscope slides with a needle so that the fibers will be uniformly distributed and not bunched together. The slides shall then be placed in an air-bath to expel the water, cooled and stained with a drop or two of zinc-chlor-iodide solution.

NOTE—The zinc-chlor-iodide solution may be prepared by dissolving 4 g. of potassium iodide and 0.1 g. of iodine in 12 cc. of water, and then adding 20 g. of zinc chloride and allowing the liquid to stand until all undissolved matter has settled out. This solution must be kept in a dark place and prepared freshly from time to time, as it loses its strength. To test out the stain, a mixture of equal parts of rag filter paper, bleached soda pulp and bleacher sulfite pulp, shall be prepared, and tested as directed. If the stain is of the correct strength, the rag fibers will show a wine-red color, the soda pulp a dark purple color, due to the thicker and more opaque fiber walls, and the sulfite pulp a light purple color due to the thinner fiber walls. If the purple color is more of a violet, then too much iodine is present and more water or zinc chloride should be added. Zinc chloride intensifies the purple color, iodine the wine-red and yellow colors, and water serves to weaken the color that predominates.

(*c*) The stained pulp shall be covered with round or square pieces of thin glass approximately ½ in. in diameter, and the excess of staining solution shall be removed from the edges with a piece of filter paper.

(*d*) The slides shall be examined under a microscope having a magnification of about 100 diameters.

(*e*) The various fibers are colored as follows:

Rag Fibers.—

Cotton and linen fibers—stained wine-red.
Wool fibers—unstained by the solution.
Jute and manila fibers—stained yellowish-brown.

Paper, Wood, Straw, etc., Fibers.—

Mechanical wood pulp, sawdust, straw, bark, etc.—stained lemon yellow.
Chemical wood pulp, (sulfite or soda), stained grayish purple to purple.

(*f*) The eye may be trained to the colors and shapes of the various fibers by examining specimens of known composition under similar conditions. In this way the fibers may readily be identified in unknown mixtures by:

(1) The colors to which they are stained.
(2) Their characteristic sizes and shapes.

(*g*) The fibers shall be counted in each field, disregarding any short ends projecting into the field or small fragments interspersed throughout it. Small particles of sizing, clay, or mineral matter should likewise be disregarded.

(*h*) Five fields shall be selected at random on each slide and the fibers counted in each. A total of 50 fields will be thus examined. The aggregate number of each variety of fiber counted in all the fields shall be calculated in percentage.

(*i*) The fibers present shall be reported in percentage ***based on their count,*** and should be generally accurate to within 5 per cent, depending upon the skill of the operator.

NOTE 1.—As the dimensions and specific gravity of the fibers have not been taken into consideration, the method is obviously inaccurate to this extent. Spence and Krauss[1] have worked out a set of factors for expressing the results into terms of weight, taking into consideration the dimensions and specific gravity of the fibers. This method is recommended, although it is somewhat slow and laborious.

NOTE 2.—As a referee method, or in case greater accuracy is desired, a separate microscope test shall be run on the three portions taken from each specimen roll. The minimum, maximum and average percentage of the various fibers present shall then be calculated.

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[1] *Paper*, Vol. XX, No. 11, p. 11, May 23, 1917.

TENTATIVE METHODS OF TESTING BITUMINOUS MASTICS, GROUTS AND LIKE MIXTURES[1]

Serial Designation: D 147 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1923, 1924, 1925.

I. DEFINITIONS.

Scope.

1. These methods apply to bituminous mastics, grouts and like mixtures, which may be classified and defined as follows:

Bituminous Grout.—A mixture of bituminous material as a binder and sandy mineral matter as an aggregate, which when heated to a suitable temperature becomes sufficiently fluid to flow into place without mechanical manipulation, and which on cooling congeals to a compact mass.

Asphalt Mastic.—(1) A mixture containing asphaltic material as a binder and graded mineral matter as an aggregate, or (2) pulverized native rock asphalt (to which asphaltic material may have been added); either of which when heated to a suitable temperature may be poured into place but which requires a trowel to form it into a compact mass.

Asphalt Mastic Cake.—A mixture containing asphaltic material as a binder and an aggregate consisting chiefly of calcareous or siliceous dust cast into the form of blocks or "cakes" and adapted for use in preparing asphalt mastic.

II. PREPARATION OF SAMPLES.

Preparation of Samples.

2. (*a*) Bituminous grouts shall be heated in an oven or on a hot plate in a pan or other suitable container at the lowest possible temperature to prevent overheating and volatilization, and when sufficiently fluid, shall be thoroughly stirred to insure a uniform sample, whereupon 10 to 30 g. shall be taken for analysis.

(*b*) Asphalt mastics or mastic cake shall be warmed on a hot plate or in a hot oven until soft enought to be broken up or stirred so that a representative sample for analysis may be taken. The amount taken for analysis will depend upon the amount of coarse gravel or stone in the mixture. The larger the gravel or stone, the larger will be the sample required for accuracy. The size of samples to be taken shall be as follows: Where all particles pass a 10-mesh sieve, 10 to 30 g.; where 25 per cent of the aggregate is retained on a 10-mesh sieve, 50 g.; where 50 per cent of the aggregate is retanied on a 10-mesh sieve, 100 g.; and where 75 per cent of the aggregate is retained on a 10-mesh sieve, 200 g.

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[1] Criticisms of these Tentative Methods are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

NOTE.—Where the properties of the extracted bituminous matter are to be determined, 500 g. shall be taken and extracted with pure benzol as described in Sections 4 and 6 and examined as described in Section 7.

III. APPARATUS.

(*A*) *For Analysis of 10 to 30-g. Samples.*

Apparatus, 10 to 30-g Samples.

3. In cases where a 10 to 30-g. sample is sufficient, the analysis shall be carried out by means of the glass extractor described in Section 8 of the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1]

(*B*) *For Analysis of 50 to 500-g. Samples.*

Apparatus, 50 to 500-g. Samples.

4. The apparatus for analysis of samples containing coarser aggregate shall be the large extractor shown in Fig. 1, consisting of a large brass cylinder, through the bottom of which projects a 16-candle-power incandescent carbon filament bulb to supply heat to the extraction apparatus proper, which is held in the upper portion of the cylinder. This apparatus is composed of a cylindrical brass vessel for holding the solvent, a cylindrical wire basket made of 80-mesh wire cloth suspended in the cylinder, and an inverted conical condenser which serves as a top.

IV. PROCEDURE.

(*A*) *Analysis of 10 to 30-g. Samples.*[2]

Procedure.

5. An ordinary Whatman or S. and S. Filter Thimble shall be dried and weighed. The weighed sample shall be placed in the thimble and 40 to 50 cc. of CS_2 poured over the sample. The thimble containing the sample shall be suspended under the condenser by a fine wire bail. The flask shall be cautiously heated by a steam-bath or electric heater just enough to vaporize the solvent. Cold water is circulated through the condenser. The heat evaporates the carbon disulfide in the flask. This condenses upon the condenser and drops back upon

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[1] 1924 Book of A.S.T.M. Standards.

[2] This method and the method for analysis of larger samples are especially adapted for asphalt mastics, grouts and mastic cake. If the binder is coal-tar pitch, the "free carbon" constituent of the binder will remain with the mineral aggregate. The committee has under consideration a possible separate method for analysis of coal-tar mastics.

the sample through which it filters, thus dissolving out the bitumen which collects in the bottom of the flask.

The extraction should be discontinued when the CS_2 drops colorless from the filter. The time of extraction will depend upon the nature of the bitumen and mineral aggregate in the sample and upon the degree of heat applied, the coldness of the water in the condenser and other factors. In some cases extraction may be complete in one hour, in others four or five hours may be necessary.

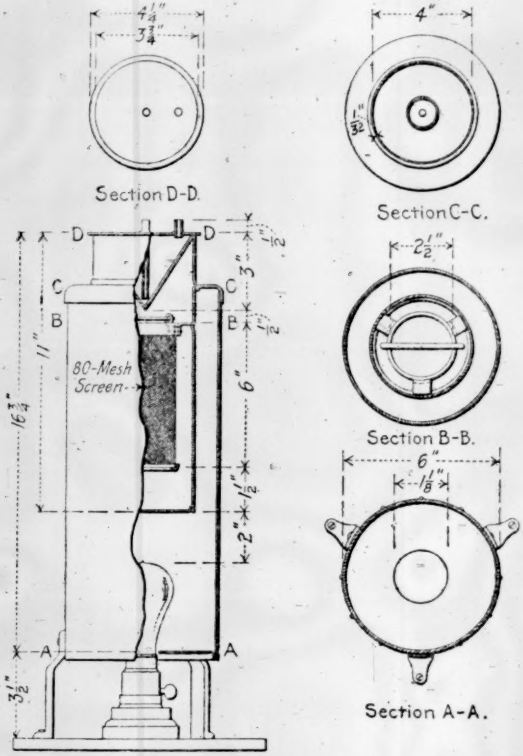

FIG. 1.

When the solvent comes through clear, the filter shall be removed and washed with a fine jet of CS_2 from a washing bottle to wash out any bitumen that may be retained at the top of the paper and to break up any channels that may have been formed by the CS_2 passing through. If the washings show any color, the thimble shall be put back and extraction continued until the solvent again becomes colorless. It shall then be removed, dried carefully, at a low temperature at first to prevent ignition of the absorbed CS_2, and finally to constant weight at 100° C. (212° F.), cooled and weighed.

The solution in the flask shall be rinsed into a weighed porcelain or silica evaporating dish or crucible and the solvent burned off under a hood. The residue shall be ignited over a flame or in a muffle and the ash weighed and the weight added to that of the mineral matter in the filter paper. This is to correct for the fine mineral matter which will be carried through the paper by the solvent. Should there be a considerable amount of ash recovered in this way, and if it is found that the mineral matter is calcium or other carbonate, it shall be recarbonated by repeated treatment with ammonium carbonate solution and finally ignited at a dull red heat. Ordinarily, however, the mineral matter going through the paper will be so small in amount that the difference caused by ignition may be neglected.

The corrected loss in weight on the original sample represents the percentage of bitumen.

The mesh analysis of the mineral aggregate shall be made in accordance with the Standard Method of Mechanical Analysis of Sand or Other Fine Highway Material, except Fine Aggregates Used in Cement Concrete (Serial Designation: D 7) of the American Society for Testing Materials.[1] The extracted residue shall now be transferred from the thimble to the 200-mesh sieve, the paper being gently rubbed to free adhering particles.

The aggregate on the sieve shall be gently rubbed with the fingers to break up lumps and to free any particles of fine dust that might adhere to larger sand particles. The sieve shall be shaken over a piece of paper from side to side with the right hand, striking it sharply against the palm of the left hand until no appreciable amount of dust comes through the sieve on to the paper. The paper shall be cleared from time to time by raising one side with the left hand and rolling off the siftings, so that it can be seen when the sifting is complete.

The material remaining on the 200-mesh sieve shall be weighed and the amount of 200-mesh material which has passed through shall be determined by difference.

This operation shall be repeated upon the coarser sieves in order and the amount passing each sieve and retained on the next finer recorded as percentage of the original sample.

(*B*) *Analysis of 50 to 500-g. Samples.*

6. A large filter paper, 12 or 13 in. in diameter, shall be fitted inside the wire basket of the extractor by folding once more than in

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[1] 1924 Book of A.S.T.M. Standards.

ordinary filtering, or by wrapping it over a form which fits inside the basket (a cylindrical bottle of proper size makes a good form) and placing it inside the basket.

The basket with contained filter paper shall be dried and weighed. The sample shall be weighed and packed in the filter paper in the basket. Care should be taken not to pack all coarse particles in one place and the fine particles in another but to have them mixed together in uniform proportions.

The sample shall be covered with a disk of felt or wad of absorbent cotton to insure even distribution of the dropping solvent, thus preventing it from forming a channel through the sample. The basket shall be suspended in the extractor and 150 to 200 cc. of CS_2 poured over the felt or cotton. The condenser shall be placed over the top and water circulated through it. Current shall be started through the electric lamp underneath the extractor and the extraction carried on exactly as in the smaller glass extractor, but on a larger scale. The time for extraction will vary from 3 to 12 or more hours, depending upon the nature of the sample.

To determine when extraction is complete, the condenser shall be raised and the basket lifted out to observe if drippings are clear. One or two drops caught upon white filter paper should leave but a light stain.

The drying and weighing of the basket, burning off of the solution for correction, and the sifting of the mineral aggregate shall be carried on exactly as in the case of the smaller sample as described in Section 5, except that in sifting mineral aggregates containing coarse stone, it is well to remove the stone by putting the mineral aggregate first through a 10-mesh sieve, as the large stone would injure the 200-mesh sieve.

The stone removed by the 10-mesh sieve may be screened through ¼-in., ½-in., and coarser screens in order, as described in the Standard Method of Mechanical Analysis of Broken Stone or Broken Slag, except Aggregates used in Cement Concrete (Serial Designation: D 18) of the American Society for Testing Materials[1] and the fine material passing the 10-mesh sieve through 200-mesh and coarser sieves in order, all amounts passing any one screen or sieve and retained on the next finer one being reported as percentage of the original sample.

Thus the ingredients of the mixture may be roughly separated by the use of the proper sized sieves, and examined for physical and chemical characteristics.

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[1] 1924 Book of A.S.T.M. Standards.

NOTE.—The siftings passing the 200-mesh sieve will consist largely of the dust or pulverized rock used in the mixture. The material between the 100-mesh and 10 mesh will consist largely of the sand used in the mixture, with any particles of fine crushed stone within these limits that existed in the original materials. Gravel or broken stone as a rule will be larger than 10 or 8 mesh.

Carbon tetrachloride, benzol, or chloroform may be used instead of carbon disulfide, with the only difference that in the case of non-inflammable solvents, the solvent will have to be evaporated from the solution of bitumen to determine the ash for correction instead of burning off directly. When the solvent is expelled, the bitumen can be ignited for ash.

(*C*) *Recovery and Examination of Extracted Bitumen.*

7. The benzol solution containing the asphalt shall be evaporated to a thick viscous liquid on a steam or water bath, then transferred to a steam oven at 105° C. (221° F.) until its weight is nearly equal to the percentage of asphalt calculated in accordance with Section 5. It shall then be transferred to a hot plate and heated carefully with stirring at a temperature not exceeding 121° C. (250° F.) until its weight is exactly that determined in accordance with Section 6.

(*D*) *Solubility of Aggregate in Hydrochloric Acid.*

8. Where the mastic is to be used for acid-proof floors, tank linings, etc., pulverized silica and other materials insoluble in acid are used in preparing the mixture. The most important chemical property, therefore, is solubility or insolubility in mineral acids. This determination may be carried out in the following manner:

About 2.5 g. of the dust, or of the coarser material, or stone which has been previously pulverized in a mortar shall be dried to constant weight at 325° F. The loss will represent any moisture that may have been retained in the material as well as any absorbed solvent that may not have been expelled while drying the sample after extraction.

A 1-g. sample of the dried material shall be placed in a 250-cc. beaker, covered with a watch glass and 10 cc. of concentrated c. p. hydrochloric acid added, diluted with 50 cc. of distilled water. Any lumps shall be broken up by means of a glass stirring rod. Any effervescence which may occur shall be noted. If considerable effervescing takes place, the sample may be said to be carbonate. If all the sample, or practically all, dissolves, the determination need not be carried farther. If apparently insoluble, it shall be heated carefully, brought to boiling for 15 minutes and filtered through a tared Gooch filter, washing the insoluble residue with hot water. It shall be ignited and weighed and the loss reported as the percentage soluble in hydrochloric acid.

TENTATIVE METHODS OF TESTING SMOOTH-SURFACED ASPHALT ROLL-ROOFING, SLATE-SURFACED ASPHALT ROLL-ROOFING AND SLATE-SURFACED ASPHALT SHINGLES[1]

Serial Designation: D 228 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. These methods cover the examination of roofings and shingles composed of roofing-felt, saturated and coated on both sides with asphalt, and surfaced on the top with either powdered or granulated mineral matter, and on the under side with powdered mineral matter.

I. SAMPLING

Sampling. 2. From each shipment or portion thereof representing a product of the same kind, class and weight, a number of rolls or bundles shall be selected at random, equivalent to one-half the cube root of the total number of rolls or bundles included in the lot, except that in lots of 1000 or less, five rolls or bundles shall be taken. If the cube root, as calculated, proves to be a fractional number, it shall be expressed as the nearest higher whole number. For convenience, the following table is given, showing the number of samples to be selected from shipments of various sizes.

Packages in Shipment	Number of Packages Selected	Packages in Shipment	Number of Packages Selected
Up to 1000	5	5 833 to 8 000	10
1001 to 1728	6	8 001 to 10 648	11
1729 to 2744	7	10 649 to 13 824	12
2745 to 4096	8	13 825 to 17 576	13
4097 to 5832	9	17 577 to 21 952	14

II. DETERMINATION OF SIZE AND WEIGHT

(A) *Smooth-roll and Slate-roll Roofing*

3. Each roll selected shall be stripped. The wrappers, packing material, nails and cement taken from each roll shall be weighed

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

together to the nearest $\frac{1}{4}$ lb. and the maximum weight thereof per roll shall be recorded. The quantity and character of nails and cement shall be recorded.

4. Each roll shall be unwound, discarding any detached surfacing that may fall off in so doing, and the workmanship and finish of the roofing observed.

5. The length and width of each roll shall be measured to the nearest $\frac{1}{4}$ in., and the square feet of material contained in each roll calculated.

6. The rolls shall be rewound, fastened with pieces of light string and then each roll shall be weighed to the nearest $\frac{1}{4}$ lb. The weight of roofing per 108 sq. ft. contained in each roll shall be calculated and the minimum weight per 108 sq. ft. recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the entire shipment.

7. The average weight per 108 sq. ft. for the rolls examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the entire shipment.

8. From the rolls examined, the one whose weight is nearest the average weight of the lot shall be selected. The first convolution or two shall be unrolled, and with a knife and straight edge, the sheet shall be cut across at right angles to the edges. A section measuring exactly 30 in. in the direction of the roll's length shall then be removed. The width of this section shall be measured to the nearest $\frac{1}{32}$ in. and weighed with an accuracy of $\frac{1}{4}$ oz. The weight in pounds per 108 sq. ft. shall then be calculated from the following formula:

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of 30-in. section in ounces}}{\text{Width of 30-in. section in inches}} \times 32.4$$

This must check within 1 per cent of the average weight per 108 sq. ft. of the shipment (Section 7). If this sample fails to do this, then additional samples shall be cut from the same roll, until one is obtained which does, which sample shall be kept for further examination.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, a 30-in. sample shall be taken and examined separately from each roll sampled in Section 2.

(*B*) *Slate-surfaced Shingles*

9. Each bundle selected shall be stripped. The packing material shall be weighed to the nearest $\frac{1}{4}$ lb., recording the weight separately for each bundle. The maximum weight of packing material per "square" shall be recorded.

10. The shingles in each bundle, *a*, shall be counted, discarding

any detached surfacing that may fall off in so doing. The workmanmanship and finish of the shingles shall be observed.

11. The shingles contained in each bundle shall be weighed to the nearest $\frac{1}{4}$ lb. (b). The dimensions of the shingles shall be accurately measured and the net area of material in each shingle calculated in square feet (c).

12. The area in square feet of material contained in each bundle ($a \times c$) shall be calculated. The weight of roofing material per 108 sq. ft. contained in each bundle $\left(\frac{b}{a \times c} \times 108\right)$ shall be calculated, and the minimum weight per 108 sq. ft. recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the entire shipment.

13. The average weight (d) per 108 sq. ft. for the bundles examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the entire shipment. Also, the average weight per shingle $\left(\frac{c \times d}{108}\right)$ shall be calculated.

14. From the bundles selected, a definite number of representative shingles, whose weight shall fall within 1 per cent of the average weight ascertained in Section 13 shall be taken for further consideration. The number of shingles so selected shall represent as closely as possible 6 sq. ft. of material, and shall be selected as far as possible from different bundles.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, then a similar sample shall be taken and examined separately from each bundle of shingles sampled in Section 2.

III. DETERMINATION OF PLIABILITY OF SMOOTH-ROLL ROOFING AND SLATE-ROLL ROOFING

15. With a photographic trimmer five strips shall be cut from the sample of roofing, each 1 in. in width and 6 in. in length, in the direction of the fiber grain, and immersed in water at 77° F. (25° C.) for 10 to 15 minutes. These strips shall be removed from the water and immediately bent through 180 deg. at uniform speed, in exactly 2 seconds, over a mandril of the following size:

Smooth-roll Roofing—At least 3 of the 5 strips shall not crack over a mandril 10 mm. in diameter.

Slate-roll Roofing—At least 3 of the 5 strips shall not crack on a mandril 25 mm. in diameter.

IV. DETERMINATION OF BEHAVIOR ON HEATING

16. Two strips shall be cut from the sample of roofing or shingles, each approximately 8 by 8 in. They shall be weighed and suspended

vertically in the center of an air-oven maintained at 176° F. (80° C.) ± 5°F. (3° C.) with a thin wire fastened through holes punctured near one edge. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The strips shall be maintained at the prescribed temperature for exactly two hours, then cooled, removed carefully and each specimen weighed. The average loss shall be calculated as a percentage. Any change in appearance of the specimen shall be recorded, such as flowing, sagging, blistering or absorption of the surface coatings; also sliding of granular surfacing.

V. DETERMINATION OF SATURANT IN MOISTURE-FREE FELT

17. Two strips shall be cut from the sample of roofing or shingles, each approximately 2 in. in width and 8 in. in length. They shall be warmed in an oven at a temperature of approximately 176° F. (80° C.). Then with a knife, the front and back coatings shall be peeled off, care being taken to remove as little as possible of the saturated felt, and to assure that all of the coatings and surfacing are stripped from the felt. The saturated felt so obtained shall be weighed. It shall be extracted with benzol, chloroform, carbon tetrachloride or carbon disulfide, in a suitable extractor and the extraction continued until the drippings have become colorless. The desaturated felt shall be removed from the extractor, placed in a tared vessel, heated in a ventilated oven at 225° F. (107° C.) for 30 minutes, cooled in a desiccator, and the felt finally weighed as rapidly as possible. The heating shall be repeated until the weight of the felt remains constant as determined by two consecutive weighings taken not less than 10 minutes apart, which shall show a further loss of not more than 0.1 per cent. From the weight so obtained and the original weight, the percentage of saturation carried by the moisture-free felt shall be computed.

VI. DETERMINATION OF WEIGHT OF MOISTURE-FREE FELT

18. Two strips shall be cut from the sample of roofing or shingles, each approximately 2 in. in width and 8 in. in length. Each of these shall be accurately weighed in grams and the length and width measured to within 1 mm. or $\frac{1}{32}$ in. It shall be extracted with benzol, chloroform, carbon tetrachloride or carbon disulfide, in a suitable extractor and the extraction continued until the drippings have become colorless. The desaturated felt shall be removed from the extractor, being careful not to lose any adhering mineral matter, placed in a tared vessel, heated in a ventilated oven at 225° F. (107° C.) for 30 minutes,

cooled in a desiccator, any adhering surfacing material brushed off and retained and the felt finally weighed as rapidly as possible. The heating shall be repeated until the weight of the felt remains constant as determined by two consecutive weighings taken not less than 10 minutes apart, which shall show a further loss of not more than 0.1 per cent. The benzol extract and accompanying mineral matter shall be retained for further examination. From the weights so obtained and the respective areas of the strips of desaturated felt, the weight of moisture-free felt shall be computed in pounds per 108 sq. ft., from the following formulas and the results averaged:

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of moisture-free felt in grams}}{\text{Area of specimen in square centimeters}} \times 221.2$$

or

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of moisture-free felt in grams}}{\text{Area of specimen in square inches}} \times 34.3$$

VII. DETERMINATION OF ASH IN MOISTURE-FREE FELT

19. The strips of desaturated felt obtained as described in Section 18 shall be cut into squares about 2 cm. on each side and thoroughly mixed. Pieces amounting to about 10 g. shall be selected at random and dried at 225° F. (107° C.) for 30 minutes. They shall be cooled in a desiccator, accurately weighed and then incinerated in a weighed crucible either over an open flame or in a muffle until all the carbon is consumed. A few drops of ammonium carbonate solution shall then be added, the sample ignited gently and weighed. The percentage of ash shall be calculated on the basis of the moisture-free felt.

VIII. DETERMINATION OF MINERAL SURFACING AND MINERAL MATTER IN COATINGS

20. The slate and other mineral matter recovered from the extraction process described in Section 18 shall be combined with the mineral matter removed from the surface of the desaturated felt and that resulting from the evaporation and ignition of the benzol extract. The mineral matter shall be dried in air free from the odor of benzol, then heated in an oven for 30 minutes at 225° F. (107° C.), and finally cooled in a desiccator and weighed. From the combined weight of the specimens taken in Section 18, the total "mineral surfacing and mineral matter in the coatings" shall be calculated in pounds per 108 sq. ft.

TENTATIVE METHODS OF TESTING INSULATING VARNISHES[1]

Serial Designation: D 115 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1922, 1923, 1924, 1925.

Material Covered.

1. These tests are intended for varnishes which are applied by brushing, dipping or spraying, and are primarily for the purpose of providing electrical insulation.

I. SPECIFIC GRAVITY.

Specific Gravity.

2. The specific gravity shall be measured with a pyknometer, Westphal balance or with a hydrometer so graduated that the specific gravity can be determined to 0.001. The temperature of the varnish shall be not less than 18° C. (64.4° F.) nor more than 22° C. (71.6° F.) and corrected to 20° C. (68° F.) by applying a correction of 0.0007 per 1° C. (0.0004 per 1° F.).

II. VISCOSITY.

Viscosity.

3. (*a*) The viscosity shall be determined with a Stormer or a MacMichael viscosimeter. For the Stormer instrument, it shall be stated in terms of the viscosity of distilled water determined with the same instrument and under the same conditions. For the MacMichael viscosimeter, the viscosity shall be stated in absolute units, the centipoise (cp.). The short-tube type of efflux viscosimeter usually employed for lubricating oils is not acceptable. The temperature of the varnish shall be 20° C. (68° F.).

(*b*) The MacMichael viscosimeter shall be calibrated as follows:

Obtain fair-sized samples (2 to 5 gal. or more) of several oils of suitable viscosities. These should be selected so that they fall in the ranges of viscosity in which measurements are likely to be made. This is to permit calibration of the instrument with an oil of the same order of viscosity as the products that are to be tested. A convenient series of samples for the testing of insulating varnish is:

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[1] Criticisms of these Tentative Methods are solicited and should be directed, preferably before January 1, 1926, to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

Mineral Oil having an absolute viscosity of approximately 20 centipoises at 20° C. (approximately 65 seconds, Saybolt Universal Viscosity, at 37.8°C.).

Mineral Oil having an absolute viscosity of approximately 40 centipoises at 20° C. (approximately 100 seconds, Saybolt Universal Viscosity, at 37.8° C.).

Mineral Oil having an absolute viscosity of approximately 170 centipoises at 20° C. (approximately 300 seconds, Saybolt Universal Viscosity, at 37.8° C.).

Castor Oil having an absolute viscosity of approximately 992 centipoises at 20° C. (approximately 110 seconds, Saybolt Universal Viscosity, at 98.9° C.).

Retain the major portion of each of these samples and send 4 to 8 oz. to the Bureau of Standards, Washington, D. C., for determination of absolute viscosity. Specify 20° C. as the temperature at which measurement are to be made. The samples, if they are properly refined filtered oils, are regarded as stable for at least one year if kept uncontaminated in closed opaque containers.

NOTE.—Standardized samples of known absolute viscosity may be obtained directly from the Bureau.

(*c*) When using the MacMichael viscosimeter it is necessary (1) to obtain the zero adjustment prior to placing the varnish in the cup, (2) to adjust to a sufficiently low speed to prevent eddy currents.

(*d*) The report shall include such details as the kind of instrument used, the size of the counterweight if a Stormer instrument is used, or the size of the wire in case the MacMichael instrument is used, etc.

III. FLASH POINT.

Procedure for Flash Point.

4. The flash point shall be determined in accordance with the Standard Method of Test for Flash Point of Volatile Flammable Liquids (Serial Designation: D 56) of the American Society for Testing Materials.[1]

IV. TIME OF DRYING.

Test Specimen.

5. (*a*) Specimens for this test shall be pieces of thoroughly cleaned, smooth sheet copper or brass about 3 cm. (1.18 in.) wide and 20 cm. (7.88 in.) long and about 0.127 mm. (0.005 in.) thick.

(*b*) The specimen shall be dipped once in the varnish at a room temperature of approximately 20° C. (68° F.) and withdrawn slowly and uniformly (about 38 cm. (15 in.) per minute). The consistency of the varnish shall be first so adjusted by trial that, when dry as determined in accordance with Section 7, the thickness of the film of varnish on each side of the metal shall be between 0.022 mm. (0.0009 in.) and 0.026 mm. (0.001 in.). Care shall be taken before dipping the specimens that the varnish has stood in the dipping tank for a sufficient length of time to be free from air bubbles.

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[1] 1924 Book of A.S.T.M. Standards.

Procedure.

6. (*a*) Specimens of air-drying varnish shall be dried in dust-free air at a room temperature of approximately 20° C. (68° F.).

(*b*) In the case of baking varnishes, six specimens shall be dipped and allowed to drain at a room temperature of approximately 20° C. (68° F.) until the varnish is set as indicated when the impression left on the surface by pressing lightly thereon with a finger will not become obliterated by further flow of the material. They are then to be dried in dust-free air in an oven at 105 to 110° C. (221 to 230° F.). At the end of the first 30 minutes, and again at the end of each 10-minute period thereafter, one specimen shall be taken from the oven and examined. In the case of slow-drying varnishes, this 10-minute period may be lengthened at the discretion of the operator.

Time of Drying.

7. The varnish shall be considered dry when a specimen will not stick to itself when folded and pressed together between the thumb and finger at a temperature of approximately 20° C. (68° F.).

V. DIELECTRIC STRENGTH TEST.

Dielectric Strength Test Specimen.

8. (*a*) Specimens for the dielectric strength test shall be prepared by dipping pieces of thoroughly cleaned, smooth sheet copper or brass about 20 cm. (7.88 in.) square and about 0.127 mm. (0.005 in.) thick into the varnish which shall be at the consistency prescribed in Section 5 (*b*).

(*b*) Each specimen shall be dipped twice, as specified in Section 5 (*b*), once in each direction, in order to give a more uniform thickness of coating. The specimen shall be dried after each dip in the same vertical position in which it was dipped.

(*c*) Specimens of air-drying varnish shall be dried in dust-free air after each dip at a room temperature of approximately 20° C. (68° F.) for a period 600 per cent longer than that determined in accordance with Section 7, provided such period does not exceed 24 hours.

(*d*) Specimens of baking varnish shall be drained and then baked in dust-free air after each dip for a period 300 per cent greater than that determined in accordance with Section 7 provided such period does not exceed 24 hours.

(*e*) The final thickness of the film of varnish on each side of the specimen shall be between 0.044 mm. (0.0018 in.) and 0.052 mm. (0.002 in.).

Procedure.

9. (*a*) The dielectric strength of the two films of varnish shall be determined by applying alternating potential to two circular metal disks, 5.08 cm. (2.0 in.) in diameter and with edges rounded to a radius of 0.64 cm. (0.25 in.) which are placed in contact with the two sides of the specimen directly opposite each other and under a pressure

of approximately 0.5 kg. (1.1 lb.). Starting at zero, the voltage shall be increased uniformly to breakdown at a rate of 0.5 kilovolt per second, except that if breakdown occurs at this rate in less than 40 seconds, the rate shall be decreased so that breakdown will occur in not less than 40 seconds. If the material fails at less than 5 kilovolts, the minimum time shall be reduced from 40 seconds to 20 seconds. Ten such punctures are to be made at various points selected at random on each specimen. In each test the thickness of the films of varnish is to be determined as close to the point of puncture as practicable.

NOTE.—When necessary, in order to get ten punctures, additional specimens should be tested.

(*b*) The frequency of the test potential shall be not greater than 100 cycles per second, and each part of the testing apparatus shall have a continuous rating of not less than 2 kva. (preferably larger). The wave form shall be a sine curve as defined, and the voltage shall be measured by methods approved by the American Institute of Electrical Engineers.[1]

(*c*) The voltage may be controlled by any approved method which does not distort the wave form beyond the limits prescribed above and which does not subdivide the voltage in steps greater than 500 volts. The apparatus shall comply with the Standards of the American Institute of Electrical Engineers.

Dielectric Strength.

10. The volts at puncture, the net thickness of insulation and the volts per mil of net thickness shall be reported for each of the ten tests together with the average maximum and minimum volts per mil.

VI. WATER ABSORPTION TEST.

Procedure.

11. Specimens similar to those described in Section 8 shall be immersed in water at a room temperature of approximately 20° C. (68° F.) for a period of 24 hours. Upon removal from the water, the surface water shall be wiped off and dielectric strength tests made immediately as described in Section 9.

Dielectric Strength.

12. The volts at puncture, the net thickness of the insulation and the volts per mil of net thickness shall be reported for each of the ten tests, together with the average, maximum and minimum volts per mil.

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[1] Standards of the American Institute of Electrical Engineers.

VII. HEAT ENDURANCE TEST.

Test Specimens. 13. For the heat endurance test, specimens shall be prepared as in Section 8. After removing not less than 1.27 cm. (0.5 in.) from one edge of the specimens, the number of strips required by Section 14 (*a*) shall be cut from the same edge, each 1.9 cm. (0.75 in.) in width.

Procedure. 14. (*a*) After setting as show by the test indicated in Section 6 (*b*), the strips referred to in Section 13 shall be placed in a uniformly heated oven in which the temperature is maintained at 100° C. (212° F.) within ± 5° C. (9° F.). A strip shall be removed at the end of 1, 2, 4, 8 and 24 hours respectively and every 24 hours thereafter. These, together with the initial strip, shall be tested as follows at a room temperature of approximately 20° C. (68° F.).

(*b*) Each strip shall be bent through 180 deg. over a rod 0.32 cm. ($\frac{1}{8}$ in.) in diameter. The number of hours of baking at which first cracking in the insulation occurs shall be noted and reported.

VIII. ACID AND ALKALI PROOF TEST.

Test Specimens. 15. The specimens to be used for the test for acid and alkali proofness shall be brass rods 1.5 cm. (0.59 in.) in diameter, 15 cm. (5.90 in.) long and carefully rounded at one end to a radius of 0.75 cm. (0.295 in.). These specimens shall be dipped three times into the varnish, leaving exposed about 3 cm. (1.18 in.) of the rod at the end opposite the rounded end. Each coat shall be dried 25 per cent longer than the period determined in Section 7. Three specimens each shall be prepared for the acid and alkali solutions.

Procedure. 16. (*a*) The three specimens shall be suspended in the acid or alkali whose effect it is desired to determine to within 3 cm. (1.18 in.) of the end of the coated portion of the rod and suitable provision made for detecting the change in the electrical resistance between the rod and the solution. (Note 1.)

(*b*) It is recommended that these tests be made in 10-per-cent solutions as follows:

Sulfuric acid of sp. gr. 1.069 at 60° F. (15.5° C.) or nitric acid of sp. gr. 1.056 at 60° F. (15.5°) C.) or hydrochloric acid of sp. gr. 1.050 at 60° F. (15.5° C.) and sodium hydroxide of sp. gr. 1.115 at 60° F. (15.5° C.).

(*c*) The temperature of the solution shall be kept at approximately 20° C. (68° F.).

NOTE 1. A simple method is to connect a voltmeter between each rod in turn and one side of a 110 volt direct current circuit, the other side of the circuit being connected to the solution through any piece of suitable metal suspended in the solution. The resistance will be inversely proportional to the deflection of the voltmeter pointer, that is, the smaller the deflection, the greater the resistance. Failure of the material will, therefore, be indicated by a sudden increase in the deflection of the voltmeter pointer.

Acid and Alkali Proofness. 17. The resistance between each rod and the solution shall be measured once per day and the number of days elapsing before breakdown occurs shall be taken as the "proofness" of the varnish.

IX. OIL PROOF TEST.

Oil Test Specimen. 18. For the test for the effect of oil, pieces cut from the specimens prepared for the dielectric strength test (Section 8) may be used after they have been punctured and measured.

Procedure. 19. The effect of oil on the varnish shall be determined by immersing the specimens in transformer oil at a temperature of 100° C. (212° F.) for 48 hours and noting the effect on the varnish as indicated, for example, by wiping with a piece of dry white cloth

NOTE.—Incipient disintegration of the surface of the varnish may sometimes be detected by examining the oil for turbidity. If a specimen of the oil filtered through filter paper can be distinguished from an unfiltered sample when the two samples are held in front of a strong light, the oil is turbid.

X. DRAINING TEST.

(ALSO KNOWN AS "WORKING VISCOSITY" TEST)

Test Specimen. 20. A strip of bond paper 0.064 mm. (2.5 mils) in thickness, 10.2 cm. (4 in.) in width and 50.8 cm. (20 in.) in length, shall be immersed in the varnish at a room temperature of approximately 20° C. (68° F.) up to a line previously drawn across the paper a few inches from the top. The paper shall be withdrawn at a slow and uniform rate (about 38 cm. (15 in.) per minute), care being taken that the varnish is free from air bubbles. The specimen shall be permitted to drain thoroughly at room temperature while suspended in a vertical position. It shall then be dried or baked (according to the type of the varnish) until dry as determined in accordance with Section 7.

Measurements. 21. The thickness of the specimen in mils shall be measured at points 5.1 cm. (2 in.), 17.8 cm. (7 in.) and 30.5 cm. (12 in.), respectively, from the line to which the specimen was immersed.

Report. 22. The thickness of each film in mils at the three points specified in Section 21 shall be recorded. The difference between the thickness at the upper point (5.1 cm.) and that at the lower point (30.5 cm.) shall be taken as a measure of the variation in the film thickness caused by draining.

XI. EVAPORATION TEST.

Procedure. 23. One hundred cubic centimeters of the varnish shall be placed in a flat-bottom crystallizing dish approximately 75 mm. (2.95 in.) in diameter and 45 mm. (1.77 in.) in height. It shall be heated to a temperature of 100° F. (37.8° C.) ± 2° F. (1.1° C.) for a period of 7 hours, the sample being exposed to still air in the open room.

Evaporation. 24. The decrease in volume of the sample shall be taken as the evaporation, this decrease being determined by noting the amount of water or kerosene that must be added to fill the dish to the original level.

NOTE.—This test is relative only. That is, it is only suitable for comparing one varnish with another when the tests are made simultaneously under exactly the same conditions.

XII. TEST FOR NON-VOLATILE MATTER.

Procedure. 25. A portion of the sample shall be placed in a stoppered bottle or weighing pipette and weighed About 1.5 g. of the sample shall be transferred to a weighed flat-bottom metal dish about 8 cm. (3.15 in.) in diameter, such as the cover of a friction-top tin can. The container shall again be weighed and the exact weight of the portion of the sample transferred to the weighed dish calculated by difference. The dish with its contents shall be heated for three hours in an oven maintained at 105 to 110° C. It shall then be weighed after cooling.

Non-Volatile Matter. 26. The ratio of the weight of the residue to that of the original sample expressed as a percentage shall be taken as the percentage of non-volatile matter in the varnish.

TENTATIVE METHOD OF TESTING ELECTRICAL INSULATING MATERIALS FOR VOLTAGE EFFECTS AT RADIO FREQUENCIES[1]

Serial Designation: D 175 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923; REVISED, 1925.

Scope.

1. This method of test is intended to determine the dielectric failure of electrical insulating materials at radio frequencies.[2]

The failure under radio frequency stress may take the form of charring, buckling, cracking, blistering, softening or chemical decomposition. Failure through the material is not abrupt and it, therefore, requires a certain amount of judgment on the part of the operator to decide just what constitutes failure in any particular case.

When a sheet of insulating material is tested between two metal electrodes passing through it, the heat generated increases with the thickness of the material, while the radiating surface remains nearly constant. Consequently, as the thickness of the sample is increased, the voltage at which failure occurs will decrease. As sufficient data are not available to allow results to be translated from one thickness to another, it is necessary that all comparative tests be made on samples of the same thickness. Hence, in the standard method given below, a definite thickness of sample is specified. This method, of course, is applicable to other thicknesses of material, but when so used the results should be compared only with similar tests made on like thicknesses of material.

I. APPARATUS

Generating Circuit.

2. Any type of generator having an output of 500 watts or more and generating continuous waves of 100 and 1000 kilocycles per second, respectively, and at voltages up to 5000 or 10,000 volts, depending upon

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

[2] A high frequency dielectric endurance test is being developed and will later be included in this tentative standard.

the class of materials tested, is satisfactory. If desired, a circuit similar to that shown in Fig. 1 may be used.

Voltmeter.

3. The voltage may be measured by means of the current through a shielded radio frequency ammeter in series with a small shielded air condenser of known capacity across the test terminals or by means of an electrostatic voltmeter designed to withstand radio frequency potentials. Suitable scales shall be provided so that reasonable accuracy will be obtained when measuring any testing voltages up to the maximum desired.

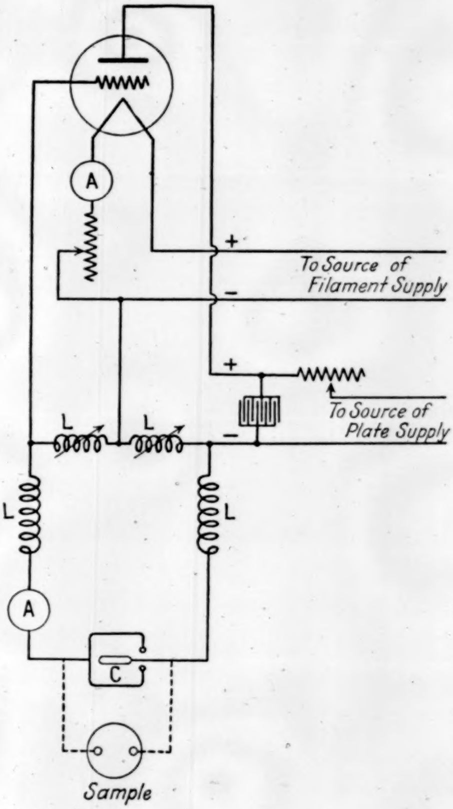

FIG. 1.—Schematic Diagram of Circuit for Determination of Voltage Effects.

If the ammeter method is used:

$$E \text{ (in volts)} = \frac{16I \times 10^7}{fC}$$

where I = current in amperes;
f = frequency in kilocycles per second;
C = capacity of air condenser in micro-microfarads.

NOTE.—In the above formula, the value 16 is an approximation, 15.92 being the exact value.

Frequency Meter. 4. The frequency meter or wave meter may be of any commercial design covering the required frequency or wave lengths and accurate to within ± 10 per cent over the wave frequency range used.

Electrodes. 5. (*a*) The electrodes shall be of brass and shall be clean and polished.

(*b*) The electrodes shall have the dimensions shown in Fig. 2.

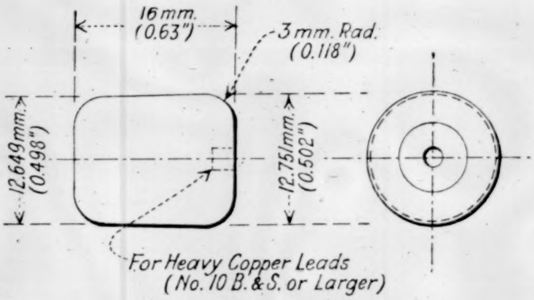

FIG. 2.—Showing Dimensions of Electrodes.

II. TEST SPECIMENS

Test Specimens. 6. (*a*) The specimens shall be representative of the material to be tested, care being taken to select material free from abnormal defects.

(*b*) The specimens shall be 6.35 mm. (0.25 in.) in thickness and may be of any convenient size or shape provided that the center electrode (see Fig. 3) is at least 57.5 mm. (2.264 in.) from the outside edge at all points. Other thicknesses may be used but the results therefrom should be used strictly for comparative purposes as explained under Section 1. If any samples are milled to reduce the thickness, care should be taken not to tear, chip or otherwise change the character of the material. Four holes 12.7 mm. (0.5 in.) in diameter, shall be provided in each specimen, one in the center and three equally spaced in a circle of 25.4 mm. (1.000 in.) radius as shown in Fig. 3.

III. PROCEDURE

Surrounding Medium and Wave Frequency.

7. (*a*) Two specimens shall be tested in the condition received at normal room temperature of about 20° C. (68° F.), one of these at 100 kilocycles per second (3000 meters wave length) and the other at 1000 kilocycles per second (300 meters wave length).

(*b*) Similarly, an additional two specimens shall be tested respectively, at the frequencies specified in Paragraph (*a*) after the specimens have been immersed in distilled water at a temperature of about 20° C. (68° F.) for 48 hours. After removing the specimens from the water, the surface water shall be wiped off with a clean absorbent cloth and the specimens then allowed to stand in the room away from drafts and direct sunlight for 5 minutes. The holes shall be drilled before immersion.

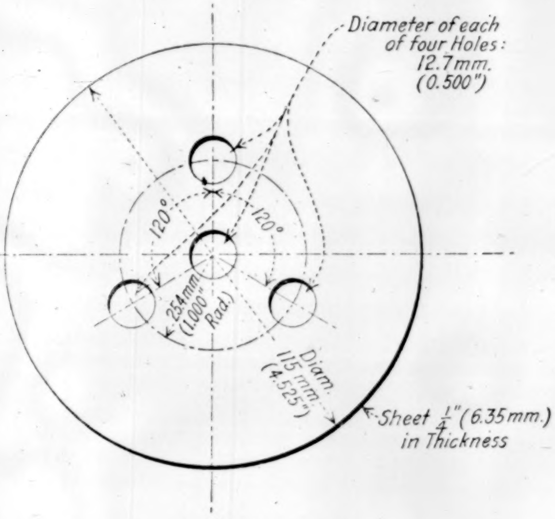
Diameter of each of four Holes: 12.7 mm. (0.500")
120°
120°
25.4mm (1.000" Rad.)
Diam. 115 mm. (4.525")
Sheet $\frac{1}{4}$" (6.35 mm.) in Thickness

FIG. 3.—Test Specimen.

Position of Electrodes in Specimens.

8. One electrode (Fig. 2) shall be inserted in the center hole in a specimen and a similar electrode in one of the other three holes. The electrodes must be a tight fit, otherwise corona may form at comparatively low voltage and destroy the material.

Connection of Electrodes to Test Terminals.

9. The electrodes shall then be connected to the test terminals of the radio-frequency generator. One side of the generator shall be grounded, and this terminal shall be connected to the center electrode of the specimen. The reason for this is that the failure generally occurs around the high potential terminal and this will allow the two subsequent tests to be made between the center and the other two holes, the center not being destroyed.

Application of Voltage. 10. For materials which ordinarily fail below 3000 volts, the voltage shall be raised in steps of 500 volts. For materials which ordinarily fail above 3000 volts, the voltage shall be raised in steps of 1000 volts. Wherever possible, the initial voltage chosen shall be such that at least three voltage steps will be required to cause failure of the material.

Test Period. 11. The voltage at each step shall be impressed for two minutes, the testing period being counted from the instant when the prescribed voltage is reached, and the time required to reach this voltage not exceeding $\frac{1}{4}$ minute. Whenever samples thicker than that specified are used, this testing period should be changed to three minutes.

Number of Tests. 12. Tests shall be made between the center hole and each of the other three holes of each specimen, the specimens being allowed to cool to room temperature after each test.

The average of these three voltages shall be considered as the dielectric strength of the specimen under the conditions of the tests.

IV. REPORT

Report. 13. The report of the tests shall include:

(*a*) The description of the material, *i. e.*, base or filler, binder, grade, color and nature of surface of specimen between electrodes;

(*b*) Room temperature and relative humidity;

(*c*) Voltage at which each dielectric failure occurs, average voltage, duration of application of final voltage, and wave frequency.

(*d*) Thickness of specimen at electrodes;

(*e*) Nature of failure.

APPENDIX

SAMPLE RECORD OF TEST

Date of Test, ... Test No., ...
Specimens submitted by, ... Number Submitted, ... Date, ...
Base or Filler, ... Binder, ... Grade, ... Color, ...
Dry Bulb, ...° C. Wet Bulb, ...° C. Relative Humidity, ...%

DIELECTRIC FAILURE TEST

	Specimen Number			
	1	2	3	4
Thickness of specimen at electrodes (mm.)				
Distance between electrode edges (mm.)				
Duration of immersion tests (hours)				
Temperature of water (deg. Cent.)				
Frequency (kilocycles per second)				
Wave length (meters)				
Initial voltage				
Voltage at failure:				
First test				
Second test				
Third test				
Duration of Application of Final Voltage (seconds):				
First test				
Second test				
Third test				
Condition of material after failure:				
First test				
Second test				
Third test				

TENTATIVE METHODS OF TESTING CABLE SPLICING AND POTHEAD COMPOUNDS[1]

Serial Designation: D 176 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923; REVISED, 1924, 1925.

Material Covered. 1. These tests are intended to cover compounds used in cable splices and potheads.

I. PHYSICAL TESTS

Physical Tests. 2. Each of the following tests shall be made in accordance with the method of test applicable, of the American Society for Testing Materials, as indicated:

(*a*) *Melting Point:* Tentative Method of Test for Melting Point of Petrolatum (Serial Designation: D 127 - 24 T).[2]

(*b*) *Softening Point,* for compounds having no definite melting point: Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36),[3] or

Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (Serial Designation: D 61).[3]

The method used should be indicated in the report.

(*c*) *Flash and Fire Points:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92).[3]

(*d*) *Evaporation:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6).[3]

(*e*) *Viscosity,* Temperature range from melting point to 150° C.: Saybolt Furol, Tentative Methods of Test for Viscosity of Petroleum Products and Lubricants (Serial Designation: D 88 - 25 T).[4]

(*f*) *Penetration Test,* Temperature range from melting or softening point to $-25°$ C.: Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5).[5]

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

[2] *Procedings,* Am. Soc. Testing Mats., Vol. 24, Part I, p. 872 (1924); also 1925 Book of A.S.T.M. Tentative Standards, p. 341.

[3] 1924 Book of A.S.T.M. Standards.

[4] See p. 707.

[5] A.S.T.M. Standards Adopted in 1925.

Brittleness.

3. To determine the brittleness of the material, the compound shall be spread on a piece of sheet metal in a layer $\frac{1}{16}$ in. in thickness. It shall then be submerged in water having a temperature at least 20° F. above the expected breaking point. It shall then be cooled 5° F. and held at this temperature for 5 minutes. The cooling shall then be continued in increments of 5° F. At each temperature interval the compounds shall be tested with the blade of a knife, inserting the point with the blade as nearly parallel with the sheet metal as is practicable, and pulling the compound from the sheet metal as rapidly as possible by rotating the blade about its back as an axis. The temperature at which the compound no longer stretches but snaps, shall be considered the breaking point.

II. DIELECTRIC STRENGTH TEST

Dielectric Strength.

4. (*a*) This test is intended to determine the dielectric strength of cable splicing and pothead compounds under voltage stresses of short duration. It is of value as a comparative test but does not indicate the permanent dielectric strength of these compounds.

(*b*) The dielectric strength of this class of material varies with the temperature. It is, therefore, recommended that a sufficient number of tests be made at different temperatures to establish the momentary dielectric strength - temperature curve from −25° C. to +100° C.

Sample.

5. A representative sample shall be taken from the original package, melted and poured directly into the testing container. Care should be used in melting and pouring the compound not to overheat it nor to entrap air. The electrodes should be heated to the approximate compound temperature before filling the container.

Electrodes.

6. (*a*) In the case of materials which are hard at room temperature, they shall be tested between hemispherical electrodes embedded therein which are $\frac{1}{2}$ in. in diameter separated by a gap of 0.1 in.

NOTE.—A convenient form of apparatus for holding the electrodes and compound is described in the Appendix.

(*b*) In the case of materials which are soft at room temperature, they shall be tested between polished brass or copper circular disk electrodes, 1 in. in diameter and having a square edge, the electrodes to be placed with their axes horizontal and coincident and with a gap of 0.1 in. between their adjacent faces.

NOTE.—This test can be made with the apparatus normally used for testing transformer and switch oils. (See the Standard Methods of Testing Transformer and Switch Oils (Serial Designation: D 117) of the American Society for Testing Materials.[1])

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[1] 1924 Book of A.S.T.M. Standards.

Testing Transformer. Voltage Control and Measurement. 7. The testing transformer used, methods of voltage control and measurement shall be as outlined in Section 2 of the Tentative Methods of Testing Sheet and Tape Insulating Materials for Dielectric Strength (Serial Designation: D 149 - 24 T) of the American Society for Testing Materials.[1]

Number of Specimens. 8. Five specimens at a given temperature shall be tested and the average value taken as the momentary dielectric strength of the compound at that temperature.

Report. 9. The report shall include the following:

(*a*) The momentary dielectric strength - temperature curve;

(*b*) The individual values of the puncturing voltage at the various temperatures.

III. COEFFICIENT OF EXPANSION

Apparatus. 10. Calibrated flasks of Pyrex or quartz glass holding approximately 250 cc. to the zero mark, having necks 1 cm. in internal diameter and graduated for 25 cc. in 0.1-cc. divisions, total capacity approximately 275 cc., shall be used.

NOTE.—If it is necessary to use a small sample, then a Cassia flask holding 100 cc., with neck graduated for 10 cc. in 0.1-cc. divisions, may be used.

11. The cylindrical oil bath for heating the sample shall be approximately 10 in. (25.4 cm.) in inside diameter and 20 in. (50.8 cm.) in inside depth with a false bottom 1 in. from the bottom and shall have provision for circulating the oil.

NOTE.—This bath may be made as follows: A shell about 10 in. (25.4 cm.) in inside diameter and 20 in. (50.8 cm.) in inside depth is electrically heated by winding the shell with resistance wire and covering with five layers of asbestos paper. A motor-droven paddle at the bottom will give effective oil circulation.

12. A lead collar to be hung on the flask during test to prevent the oil currents of the bath from moving the flask.

Calibration. 13. The capacity of the flask at the zero point and several points on the scale shall be determined by filling the flask with distilled water at a known temperature and weighing.

Procedure. 14. The flask shall first be warmed slightly, then partly filled with melted compound and placed in the bath which shall then be brought to the highest temperature of the range over which the test is to extend. The flask shall then be filled to within the last cc. marked on the neck. The bath shall be held at this temperature for not less than 30 minutes (principally to remove air bubbles) and the whole shall then be slowly cooled to room temperature (10 to 12 hours.) Before starting the test, the flasks shall be examined for the

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol., 24, Part I, p. 1039 (1924); also 1925 Book of A.S.T.M. Tentative Standards, p. 650.

the presence of cavities or irregular contraction of the compound. Some compounds, after cooling below the liquid state, tend to stick to the sides of the neck of the flask. In that case it is necessary to gradually warm the neck and flow the compound to meet the rest, after which the flask is placed in the bath for an hour or so to insure equalizing the temperature.

With the compound satisfactorily placed in the flasks at the lowest temperature, the height of the column in the neck shall be read and the bath then slowly heated. Readings shall be taken at 5° C. intervals, holding the bath as constant as possible at each point until no more expansion occurs at that point, the procedure being repeated for each point until maximum temperature is reached.

Precautions.

15. During the test, temperature readings shall be taken at top and bottom of the bath to detect any variation.

Until the compound is completely liquefied, intervals between readings should be three to four hours, especially between 35 and 55° C.

After becoming liquefied, the time necessary for obtaining a reading may be reduced to about 30 minutes.

Report.

16. (*a*) After securing the readings over the temperature range desired, a curve shall be plotted from the temperature and flask readings from which the coefficient of expansion shall be calculated from the formula:

$$\frac{V_1 - V}{(T_1 - T)\,V} + C$$

where V = original volume;
V_1 = volume at higher temperature;
T = original temperature;
T_1 = higher temperature;
C = constant = coefficient of expansion of glass container.

(*b*) The coefficient shall be calculated for three ranges as follows:

(1) From the minimum temperature at which the measurement was made to 10° C. below the melting point. This is intended to give the average coefficient for the solid condition.

(2) From 5° C. above the melting point to 100° C. This is intended to give the average coefficient for the liquid condition.

(3) From the minimum temperature at which a measurement was made to 100° C.

In reporting the coefficient of expansion the corresponding temperature range shall be indicated in each case.

APPENDIX

Because of the great difficulty in removing most cable splicing and pot head compounds from the container and from the electrodes, an arrangement was devised sufficiently inexpensive to permit of its being discarded wholly or in part after the test. That is, each specimen with its container can be entirely discarded without undue expense.

One form of container consists of a small rectangular box about 2½ in. (6.4 cm.) in length and 1½ in. (3.8 cm.) square, the ends of which are dry wood about ¼ in. in thickness. The sides and bottom are formed from a piece of thin, hard fiber tacked to the wooden end pieces. The fiber is partially cut at the corners which permits making a sharp, square bend.

Each electrode is mounted on the end of a piece of straight copper wire, No. 10 A.W.G., which passes through a hole at the center of the wooden end pieces. These holes are drilled in one operation so that they are in the same straight line. They are made slightly smaller so that the rods fit tightly and are therefore not readily moved.

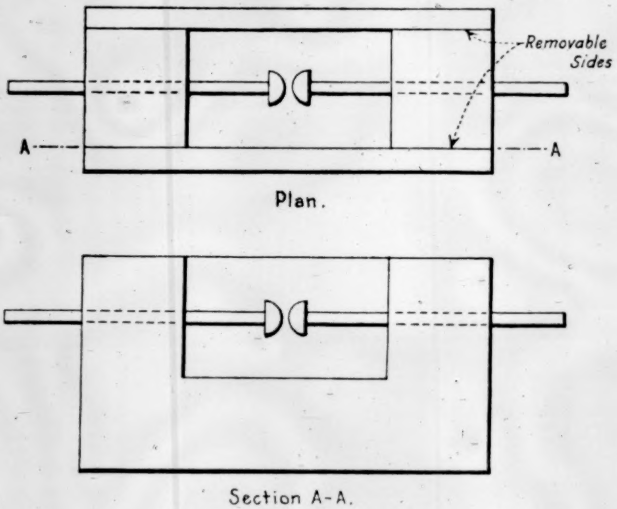

Fig. 1.—Container, Showing Electrodes in Place for Dielectric Test of Cable Splicing and Pothead Compounds.

Fig. 1 shows a sketch of a container in which the ends and the bottom are formed from one block of thoroughly dried wood which has been boiled in paraffin, the sides being pieces of thin, hard fiber tacked to the wood.

In the construction of these containers, it is obviously important that the electrodes be reasonably co-axial and that the gap remain constant.

In making tests at high voltage it will probably be found necessary to immerse the container with its specimen in oil in order to prevent flash-over between the exposed ends of the electrode rods. The ends of the rods can be bent to form hooks, by means of which the specimen holder can be readily suspended in a jar of oil.

TENTATIVE METHODS OF TESTING UNTREATED INSULATING PAPER[1]

Serial Designation: D 202 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924; Revised, 1925.

Material Covered. 1. These methods apply to untreated paper which is to be used as an electrical insulator or as a constituent of a composite material used for electrical insulating purposes.

I. SAMPLING

Sampling. 2. (*a*) From shipments consisting of rolls, a sample of at least 1 sq. yd. in area, cut across the entire width of the roll, shall be taken from every tenth roll in the shipment. From other shipments such as sheets or pads of tape, samples of equivalent area shall be taken in such a manner as to be equally representative of the shipment. In no case shall less than three samples be taken.

Note.—It is recommended that several turns of paper from each roll sampled be torn off and discarded before the samples are taken.

(*b*) The tests for physical properties shall be made upon each sample.

(*c*) The tests for acidity,[2] moisture content, size,[2] ash and for identification of fibers[2] shall be made in duplicate upon portions taken from a composite sample. This composite sample shall be made up of a sufficient number of small pieces, each of approximately 6.5 sq.cm. (1 sq. in.) in area taken in nearly equal amounts from each of the samples taken in accordance with Paragraph (*a*).

(*d*) The required number of samples shall be taken at equal intervals across the sheet, the two end samples being taken at the edges of the sheet after removing any damaged paper. When it is not possible to secure the required number of specimens in this manner in going once across the sheet, the selection across the sheet shall be repeated as many times as necessary until the required number of specimens is secured.

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

[2] The tests for acidity, size, identification of fibers, and folding endurance do not appear in these methods but will be included as soon as the tests are developed by the committee.

II. CONDITIONING SAMPLES

Conditioning. 3. Samples shall be conditioned in air maintained at a relative humidity between 60 and 65 per cent as measured with a sling psychrometer or its equivalent. The temperature of the air shall be maintained as constant as possible at some temperature between the limits of 20 and 30° C. (68 and 86° F.). The samples should remain in the conditioned air for not less than 4 hours prior to the tests and should be supported so as to allow a free circulation around each sample. The following physical tests shall be made in the conditioned air: tensile breaking strength, tearing strength, bursting strength, folding endurance,[1] air permeability,[1] and thickness.

III. MOISTURE CONTENT

Apparatus. 4. A weighing bottle, evaporating dish, thermometer, constant-temperature oven, chemical balance and desiccator are necessary for the test. The weighing bottle should be of convenient size, about 65 mm. (2.56 in.) in height and 45 mm. (1.77 in.) in diameter with a wide mouth provided with a ground-glass stopper. The chemical balance should be sensitive to 0.1 mg.

Specimen. 5. The moisture content figure is used to calculate the percentage of acidity, size, and ash of the paper to the bone-dry basis. It shall be determined on 2 to 5-g. samples selected in accordance with Section 2 (*c*).

If the moisture content of the paper as received at the purchaser's works is desired or at any other stated time, it shall be made on the sample taken at the definitely stated time by cutting small pieces of approximately 6.5 sq. cm. (1 sq. in.) in area from the required number of rolls or pads of tape in accordance with Section 2 (*a*) and immediately placed into an air-tight container.

NOTE.—Since paper adjusts itself, as regards moisture content, to the humidity of the surrounding atmosphere in a very short time (2 or 3 minutes), special care must be exercised to transfer rapidly the sample from the roll or bundle to the container and from the container to the weighing bottle.

Procedure. 6. The sample before drying shall be weighed in the tared bottle with the stopper in place. The bottle shall then be placed in the oven at 100 to 105° C. (212 to 221° F.), the stopper removed and laid alongside of the bottle and the contents transferred to the drying dish. After one hour, while still in the oven, the sample shall be replaced in the weighing bottle, and the bottle stoppered and transferred to the desiccator. The stopper should be removed while the bottle cools. When the specimen and the bottle have cooled to room temperature, the stopper shall be replaced and the bottle with its contents weighed. This process shall be repeated at intervals of one hour until the difference in weight between two successive weighings is not more than 0.2 per cent of the weight of the specimen.

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[1] The tests for acidity, size, identification of fibers, and folding endurance do not appear in these methods but will be included as soon as the tests are developed by the committee.

NOTE.—The weighing bottle or sample should not be touched with the fingers during this test.

Report.

7. The moisture content shall be expressed:

(*a*) as a percentage of the weight of the dry sample, and

(*b*) as a percentage of the weight of the undried sample.

IV. THICKNESS

Apparatus.

8. A micrometer of the spring actuated, dial type shall be used. The plunger shall be capable of being raised by the application of an upward pressure to it. The plunger surface shall be circular in shape and 12.7 mm. (0.50 in.) in diameter.

In addition to these requirements, the instrument shall conform to the following:

(*a*) The pressure applied to the plunger necessary to move the pointer from zero to a positive reading shall be not greater than 1135 g. (40 oz.).

(*b*) The pressure applied to the plunger necessary to just prevent movement of the pointer from zero to a lower reading shall be not less than 455 g. (16 oz.).

(*c*) If the instrument is provided with a device for controlling the speed of descent of the plunger, the latter shall not strike the anvil with an impact pressure greater than 1135 g. (40 oz.).

(*d*) The diameter of the dial shall be approximately 15 cm. (6 in.) and shall be graduated preferably in divisions indicating a thickness of 0.0127 mm. (0.0005 in.) and in no case greater than 0.0254 mm. (0.001 in.). Graduations indicating a thickness of 0.0254 mm. (0.001 in.) shall be at least 3 mm. (0.12 in.) apart.

(*e*) Measurements made on standard steel thickness gages shall be within the following tolerances:

INTERVALS	PERMISSIBLE DEVIATION OF READING FROM ACTUAL THICKNESS OF STANDARD STEEL GAGE
0 to 0.25 mm. (0 to 0.01 in.) incl.	±0.0025 mm. (0.0001 in.)
Over 0.25 mm. to 1.02 mm. (0.01 in. to 0.04 in.) incl.	±0.0051 mm. (0.0002 in.)
Over 1.02 mm. to 3.05 mm. (0.04 in. to 0.12 in.) incl.	±0.0102 mm. (0.0004 in.)

(*f*) Convenient means shall be provided for setting the pointer to the zero position.

(*g*) The micrometer shall be tested periodically as outlined above and if it does not conform to these requirements its mechanism should be readjusted.

Specimens. 9. Specimens shall consist of the original samples obtained in accordance with Section 2 (*a*).

Methods. 10. At least five readings shall be taken at regular intervals across the entire width of each specimen. When measuring paper less than 0.05 mm. (0.002 in.) in thickness, a sufficient number of layers to give a reading on the scale of not less than 0.13 mm. (0.005 in.) shall be used.

Report. 11. The average, maximum and minimum thicknesses obtained on each specimen shall be reported.

V. TENSILE BREAKING STRENGTH

Apparatus. 12. (*a*) A testing machine suitably designed for testing paper shall be used. The machine shall preferably be power driven.

(*b*) The capacity of the machine shall not exceed 113 kg. (250 lb.).

(*c*) The machine shall be graduated to read 1 lb. or 1 kg. or less per scale division for testing paper breaking at 22.7 kg. (50 lb.) or over, and to 0.5 lb. or 0.5 kg. or less for testing paper breaking under 22.7 kg. (50 lb.).

Specimens. 13. From each of the samples selected in accordance with Section 2 (*a*), specimens shall be cut at least ten in the machine direction and if practicable, ten in the cross-machine direction. The specimens shall not exceed 2.54 cm. (1 in.) in width and shall be 25.4 cm. (10 in.) in length with clean-cut edges.

Procedure. 14. (*a*) The ratio of the clearance distance between jaws to the width of the specimen shall be not less than 5:1 nor more than 10:1.

(*b*) The rate of travel of the movable jaw shall be constant. It shall preferably be 30.5 cm. (12 in.) per minute, but it may be within the limits of 28 cm. (11 in.) and 33 cm. (13 in.) per minute provided it is constant.

(*c*) All readings obtained when the paper breaks at or in the jaws shall be rejected.

Report. 15. (*a*) The results of the machine-direction specimens and the cross-machine-direction specimens shall be reported separately.

(*b*) The results shall be reported in kilograms or pounds together with the width of the specimen in centimeters or inches and also the average thickness.

(*c*) The maximum, minimum, and average breaking load shall be reported for the machine-direction and the cross-machine direction.

VI. TEARING STRENGTH

16. The testing machine shall be of the pendulum impulse type so designed as to produce a tear approximately 4.2 cm. (1.66 in.) long. The knife mounted on the machine to cut the slit for the tear shall be maintained sharp. **Apparatus.**

17. The specimens shall be taken from the original samples obtained in accordance with Section 2 (*b*) and shall be cut 6.3 cm. (2.5 in.) in width and at least 6.3 cm. (2.5 in.) in length. Enough specimens shall be cut so that at least five readings in the machine direction and five readings in the cross-direction, if possible, can be obtained for each original sample. **Specimen.**

18. Enough paper shall be torn so that the readings on the machine shall be not less than 10 nor more than 20 g., unless one paper alone yields a reading greater than 20 g., in which case only one paper shall be torn at one time. Readings obtained when a tear deviates more than 6.3 mm. (0.25 in.) from a straight line, shall be rejected. The readings obtained shall be multiplied by the instrument constant corresponding to the number of sheets torn. **Method.**

19. (*a*) The tearing strengths obtained on the machine-direction specimens and on the cross-direction specimens shall be reported separately in grams. **Report.**

(*b*) The average, the maximum, and the minimum tearing strengths shall be reported for the machine-direction specimens. If possible, similar results shall be reported for the cross-direction specimens.

VII. BURSTING STRENGTH

20. The testing machine shall have a circular flexible diaphragm 6.44 sq. cm. (1 sq. in.) in area. The pressure chamber shall be filled with glycerin or other suitable pressure medium and shall contain no air spaces. The test specimen shall be held in position over the diaphragm in a clamp having a circular hole approximately 6.44 sq. cm. (1 sq. in.) in area so that the diaphragm will force the paper into the hole when pressure is applied under the diaphragm. The pressure exerted on the diaphragm shall be indicated on a dial, graduated to at least 0.23 kg. (0.5 lb.) for papers giving bursting strengths 18 kg. (40 lb.) or under. A dial graduated 0.45 kg. (1 lb.) may be used in testing papers which give higher bursting strengths. The machine may be either hand-operated or power-driven, the latter being preferable. **Apparatus.**

21. If practicable, the specimen shall be so cut from the stock as to permit ten bursting tests on a line across the sheet or roll. **Specimen.**

Procedure. 22. Ten bursts shall be made. The testing machine shall be driven at a uniform speed of 120 r.p.m. until the specimen bursts.

Report. 23. The report shall include the average, the maximum, and the minimum results obtained.

VIII. ABSORPTION

(RISE OF WATER)

Apparatus. 24. A suitable container and support for specimens and scale is shown in Fig. 1.

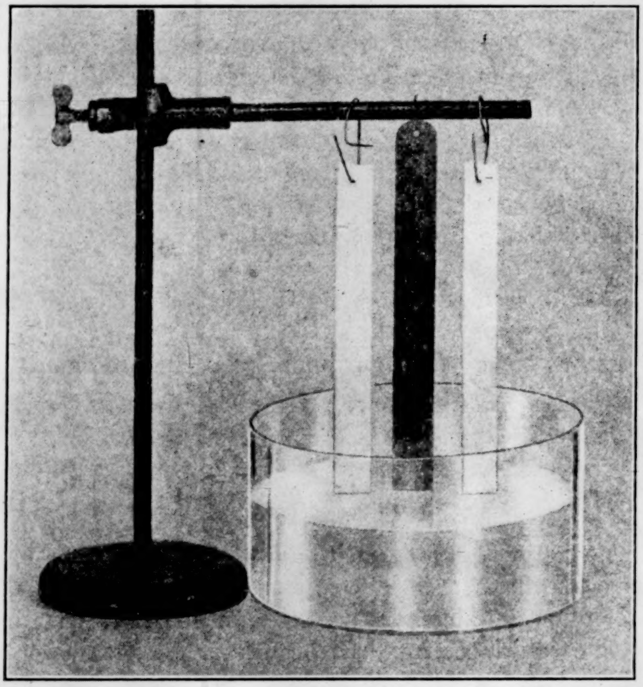

FIG. 1.—Apparatus for Absorption Test.

Specimens. 25. Ten strips 2.5 cm. (1 in.) in width and at least 12.5 cm. (5 in.) in length shall be cut from the samples obtained in accordance with Section 2 (*a*), five being cut parallel with the machine direction of the paper and five being cut parallel with the cross-direction of the paper.

Procedure. 26. The specimens shall be suspended vertically with one end dipping 3.2 mm. (0.125 in.) in distilled water at room temperature.

After five minutes of suspension, the rise of the water in the paper above the water level shall be noted. The rise may be measured by reading directly from the scale the height of absorption.

27. The rise of water shall be reported in millimeters, to the nearest 3 mm. or in inches to the nearest $\frac{1}{8}$ in. **Report.**

IX. MIL WEIGHT PER REAM
(APPARENT DENSITY)

28. A scale, sharp knife, chemical balance and weighing bottle are necessary for the test. **Apparatus.**

29. Duplicate specimens shall be taken from as widely separated portions as possible from each of the samples obtained in accordance with Section 2 (*a*) and conditioned in accordance with Section 3. **Specimens.**

30. (*a*) Each specimen while in the conditioned atmosphere (see Section 3) shall be cut accurately to any convenient size, for example, 232.3 sq. cm. (36 sq. in.) in area, and accurately weighed. Specimens of sufficient size to weigh at least from 3 to 5 g. (0.11 to 0.18 oz.) should be taken. If it is not possible to do the weighing in the conditioned atmosphere, the specimen shall be placed in a weighing bottle and tightly stoppered with a ground glass stopper before being removed from the conditioned atmosphere. The thickness of each specimen shall be determined in accordance with Section 10. **Procedure.**

(*b*) *Mil Weight per Ream of Any Size.*—The mil (or point) weight in pounds per ream of any size may be calculated from the following formula:

$$W = 0.002205\frac{mSN}{ta}$$

where m = the weight of the specimen expressed in grams;
t = the average thickness of the specimen expressed in mils (thousandths of an inch);
a = the area of the specimen expressed in square inches;
S = the area of one sheet in the basic ream expressed in square inches;
N = the number of sheets in the ream.

NOTE.—0.002205 is contracted from 0.00220462.

NOTE.—The following formulas may be used to calculate the mil weight in pounds per ream for reams of particular sizes, provided a specimen of exactly 232.3 sq. cm. (36 sq. in.) is used:

Ream of 24 by 36 in., 480 sheets.

$$W = 25.40\ \frac{m}{t}$$

where m = the weight of specimen expressed in grams;
t = the average thickness of the specimen in mils (thousandths of an inch).

25.40 is contracted from 25.3972224.

Ream of 24 by 36 in., 500 sheets.

$$W = 26.45 \frac{m}{t}$$

where m and t are the same as above and 26.45 is contracted from 26.45544.

NOTE.—The ream weight may be obtained from the above formulas by omitting the thickness "t".

(*c*) *Weight per Unit Volume.*—The weight in pounds per cubic foot (density) may be calculated from the following formula:

$$D = 3810 \frac{m}{ta}$$

Where m = the weight of the specimen expressed in grams.

t = the average thickness of the specimen expressed in mils (thousandths of an inch).

a = the area of the specimen expressed in square inches.

NOTE.—3810 is contracted from 3809.58336.

NOTE.—To convert weight in pounds per cubic foot to mil weight in pounds per ream of 24 by 36 in., 480 sheets, multiply by 0.24; to convert to mil weight in pounds per ream of 24 by 36 in., 500 sheets, multiply by 0.25.

Report. 31. The weight in pounds per cubic foot or the mil weight per ream shall be reported. The size of the ream must be given when the mil weight per ream is reported.

X. AIR RESISTANCE

Apparatus. 32. (*a*) The instrument shall consist of two aluminum open-top cylinders, one of which is inverted and slides into the other which is fixed. The movable cylinder shall be provided with a circular aperture in the closed end and a flat ring clamp for holding the paper specimen across this aperture.

(*b*) The fixed cylinder shall be 25.4 cm. (10 in.) in height and shall have an external diameter of 8.60 cm. (3.38 in.) and an internal diameter of 8.25 cm. (3.25 in.). Four slender bars, each 18.5 cm. (7.3 in.) long, 3 mm. (0.12 in.) wide and approximately 1.5 mm. (0.06 in.) thick shall be mounted vertically and equi-distantly on the inner surface of the fixed cylinder to act as guide tracks for the movable cylinder. The movable cylinder shall be graduated in units of 50 cc. and shall have a total range of 350 cc. It shall be 25.4 cm. (10 in.) high and shall have an external diameter of 7.62 cm. (3.00 in.) and an internal diameter of 7.35 cm. (2.90 in.). It shall weigh, including the flat ring clamp and the two knurled nuts, 567 g. (20 oz.) ± 5 g. (0.18 oz.). The movable cylinder and the flat ring clamp shall have a concentric circular aperture of 6.44 sq. cm. (1.00 sq. in.) in area.

When this aperture is too large for the specimen a similar movable cylinder and flat ring clamp having a concentric circular aperture of 1.61 sq. cm. (0.25 sq. in.) in area shall be used.

NOTE.—Results obtained with apertures of different areas are not directly proportional to the areas of the apertures. The proportionality factor must be determined by experiment.

Specimen. 33. The specimens shall be not less than 3.5 cm. (1.36 in.) nor more than 5.1 cm. (2.0 in.) in width cut from the original samples, taken according to Section 2 (*a*) and shall be as long as the width of the original roll of paper. If the specimens are selected from pads of tape they shall be not less than 2.1 cm. (0.81 in.) nor more than 5.1 cm. (2.0 in.) wide and at least 30 cm. (12 in.) long. There shall be as many specimens as there are original samples.

Procedure. 34. The fixed cylinder shall be placed on a rigid support so that its sides are vertical. Ethylene glycol shall be placed in it to a depth of 12.7 cm. (5 in.). The specimen (one thickness only) shall be secured tightly under the clamp on the movable cylinder completely covering the aperture. The movable cylinder is then floated on the ethylene glycol. The time required for the displacement of a certain amount of air is noted with a stop-watch. If possible, the amount of air displaced should be such that the time of displacement is not less than 20 seconds. The apparatus with its content of ethylene glycol shall be at the temperature of the conditioning room when the readings are taken. The time in seconds required for the displacement of 100 cc. (6.1 cu. in.) through a circular area (one side only) of 6.44 sq. cm. (1 sq. in.) of the paper is known as the air resistance of the paper.

NOTE.—The clamp shall be tested for leakage by substituting a piece of tin foil 0.05 mm. (0.002 in.) in thickness for the paper and testing in the manner described above. When so tested, the leakage shall not exceed the rate of 50 cc. in five hours.

The proper procedure for clamping the specimen or tinfoil is to turn both knurled nuts down onto the clamp simultaneously. If only one nut at a time is turned down, the clamp will not fit flat on the specimen and will consequently have an avoidable leak.

Ethylene glycol is used in preference to distilled water because it does not corrode aluminum whereas distilled water does.

Report. 35. The report shall include (1) the number of seconds required for the displacement of 100 cc. (6.1 cu. in.) of air; (2) the area of paper through which the air was displaced; (3) the thickness of the paper as obtained under Section 11 and (4) the room temperature.

XI. ASH

Apparatus. 36. A suitable crucible such as platinum, nickel or porcelain, a balance sensitive to 0.1 mg., and a desiccator are necessary for the test.

Specimen. 37. Not less than 2 g. of dry, finely-divided paper from the "moisture determination" sample as obtained in accordance with Section 6 shall be used.

Procedure. 38. The sample shall be transferred rapidly from the weighing bottle containing the dried sample into the crucible. The weighing bottle shall immediately be re-stoppered and re-weighed, and the exact weight of the sample obtained by taking the difference in weights of the weighing bottle and the sample. The sample shall be completely ignited in the crucible, preferably in a muffle furnace, all precautions being taken to prevent loss of ash or sample. The residue of incombustible mineral matter (ash) shall be desiccated, cooled and weighed, and the amount computed as a percentage of the weight of the dried sample.

Report. 39. The amount of ash shall be reported as a percentage of the weight of the dry sample (see Section 7 (*a*)) and also as a percentage of the weight of the undried sample (see Section 7 (*b*)).

TENTATIVE METHODS OF TESTING LAMINATED SHEET INSULATING MATERIALS[1]

Serial Designation: D 229 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

1. These tests apply to laminated flat sheet materials intended for use as electrical insulation.[2] **Scope.**

2. In referring to the cutting of the specimens and the application of the load, the following definitions apply: **Definition of Terms.**

Flatwise.—Load applied to the face of the laminations.

Edgewise.—Load applied to the edge of the laminations.

Lengthwise.—In the direction of the length of the sheet.

Crosswise.—In the direction at right angles to the length of the sheet.

NOTE.—When the sheet has the same length and width, one dimension shall arbitrarily be designated as the length, and the other as the width.

I. TENSILE STRENGTH

3. Any standard type of testing machine may be used. The machine shall be accurate to within 1 per cent on the lowest load for which it is used. Jaws which tighten under load, such as wedge grip jaws, shall be used with the specimen properly aligned. **Apparatus.**

4. Tension test specimens shall conform to the dimensions shown in Fig. 1. The surfaces of the specimen shall be smooth and free from scratches. **Specimens**

5. Five specimens cut lengthwise and five specimens cut crosswise of the sheet shall be tested. **Procedure.**

6. All tests shall be made at normal room temperature of about 20° C. (68° F.). **Temperature.**

7. The cross-head speed of the testing machine shall be such that the beam of the machine can be kept balanced, but shall not exceed 0.05 in. per minute when the machine is running idle. **Speed of Testing Machine.**

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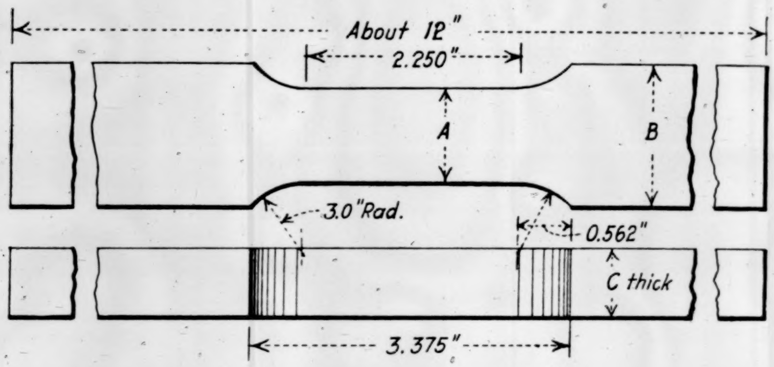
[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

[2] Tests for shearing strength, compressive strength, moisture absorption, oil absorption and degree of curing are being studied by the committee. Various mechanical tests, particularly applicable to thin laminated sheet materials, are being developed.

8. When it is desired to obtain the modulus of elasticity, a suitable extensometer measuring the elongation on a 2-in. gage length shall be used. The cross-head speed of the testing machine shall not exceed 0.03 in. per minute when the machine is running idle.

9. Strain readings shall be taken by increments of load not exceeding about 1000 lb. per sq. in. The modulus shall be calculated from the total change of length between zero load and stress of 5000 lb. per sq. in.

NOTE.—For some materials the stress strain curve will not form a straight line, but will be a continuous curve from the origin to the breaking load.



When C is less than $\frac{1}{8}$ in., A is $\frac{1}{2}$ in., B is $\frac{3}{4}$ in.
When C is over $\frac{1}{8}$ in., or over, A is 1 in., B is $1\frac{1}{2}$ in.

FIG. 1.—Tension Test Specimen for Laminated Sheet Insulating Materials.

Report. 10. The report shall include:

(*a*) The direction in which the specimen was taken, whether lengthwise or crosswise of the sheet.
(*b*) The thickness and width of each specimen in inches or centimeters.
(*c*) The breaking load of each specimen, in pounds or kilograms.
(*d*) The tensile strength of each specimen, in pounds per square inch or in kilograms per square centimeter.
(*e*) The location and nature of the fracture.
(*f*) The modulus of elasticity, if determined.
(*g*) The room temperature.

II. TRANSVERSE STRENGTH

(*A*) *For Materials ½ in. Thick or Over*[1]

Apparatus. 11. (*a*) Any standard type of testing machine may be used. The machine shall have an accuracy of at least 1 per cent of the lowest load for which it is used.

(*b*) The distance between points of support shall be 4 in. for specimens ½ in. in thickness and proportionately longer for greater thicknesses. The supports shall have the contact edges rounded to a radius of ⅛ in. The load shall be applied midway between the supports by a pressure piece, the bearing edge of which shall be rounded to a radius of ⅛ in.

Specimen. 12. The test specimen shall be 5 in. in length, ½ in. in width and ½ in. in thickness, for sheets ½ in. in thickness, and for thicker sheets it shall be the full thickness of the sheet with width and length increased in proportion.

Procedure. 13. The specimens shall be tested as follows:

(*a*) Five specimens cut lengthwise of sheet, tested flatwise.
(*b*) Five specimens cut lengthwise of sheet, tested edgewise.
(*c*) Five specimens cut crosswise of sheet, tested flatwise.
(*d*) Five specimens cut crosswise of sheet, tested edgewise.

Temperature of Tests. 14. All tests shall be made at normal room temperature of about 20° C. (68° F.).

Speed of Testing Machine. 15. The cross-head speed of the testing machine shall be such that the beam of the machine can be kept balanced, but shall not exceed 0.05 in. per minute when the machine is running idle.

16. The deflection at the center in inches or millimeters shall be taken at a beam load corresponding to a calculated maximum fiber stress of 10,000 lb. per sq. in. (705 kg. per sq. cm.).

Report. 17. The report shall include:

(*a*) The directions of cutting and loading of the specimen as specified in Section 13.

(*b*) The thickness and width of each specimen in inches or millimeters.

(*c*) The breaking load of each specimen in pounds or kilograms.

(*d*) The maximum fiber stress in pounds per square inch or kilograms per square centimeter calculated from the formula:

$$S=\frac{3\ Pl}{2\ bd^2}$$

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[1] A method of test for thinner materials is being considered.

where S = maximum fiber stress.
P = breaking load in pounds or kilograms;
l = distance between supports in inches or centimeters;
b = width of specimen in inches or centimeters;
d = depth of specimen in inches or centimeters.

(*e*) The room temperature.

(*f*) The deflection at the center in inches or millimeters at a beam load corresponding to a calculated maximum fiber stress of 10,000 lb. per sq. in. (705 kg. per sq. cm.), if determined.

III. DIELECTRIC STRENGTH

18. The dielectric strength shall be determined in accordance with the Tentative Methods of Testing Sheet and Tape Insulating Materials for Dielectric Strength (Serial Designation: D 149 – 24 T) of the American Society for Testing Materials.[1]

IV. PHASE DIFFERENCE (POWER FACTOR) AND DIELECTRIC CONSTANT AT RADIO FREQUENCIES

19. The phase difference and dielectric constant shall be determined in accordance with the Tentative Methods of Testing Electrical Insulating Materials for Phase Difference (Power Factor) and Dielectric Constant at Radio Frequencies (Serial Designation: D 150 – 23 T) of the American Society for Testing Materials.[2]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 1039 (1924); also 1925 Book of A.S.T.M. Tentative Standards, p. 650.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 822 (1923); also 1925 Book of A.S.T.M. Tentative Standards, p. 657.

TENTATIVE SPECIFICATIONS

FOR

TEXTILE TESTING MACHINES[1]

Serial Designation: D 76 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to **annual revision.**

ISSUED, 1920; REVISED, 1922, 1925.

Type of Machine. 1. Textile testing machines shall be of the inclination balance or pendulum type.

Angle of Pendulum. 2. The maximum angle of swing of the pendulum in textile testing machines shall be 45 deg. from the vertical.

Pendulum Drum. 3. The minimum diameter of drum for transferring the pull on the specimen to the swinging pendulum shall be 2 in.

Testing Machine Capacity. 4. In selecting the proper capacity of a textile testing machine for a given sample of fabric or yarn, the maximum capacity of the machine shall not exceed that at which the pendulum reaches a swing of 45 deg. from the vertical. The minimum capacity of the machine when used for a given sample of fabric or yarn shall not be less than 20 per cent of the above maximum capacity.

Type of Jaw. 5. (*a*) *Fabric Jaws.*—The clamps of textile testing machines for use upon fabrics shall consist of flat metallic jaws pressing directly against the specimen. One gripping surface shall be hinged or swiveled and the other shall be rigidly connected to the frame of the jaw. The pressure between the jaws shall be secured by any suitable mechanical device so constructed as to grip the fabric firmly before the testing load is applied and to prevent visible slippage during the progress of the test.

(*b*) *Skein Jaws.*—The drums of testing machines for yarn skeins shall consist of cylindrical spools not less than 1 in. in diameter and not less than 1 in. in width, so supported that at least one shall turn freely upon its axis.

(*c*) *Individual Strand Jaws.*—The jaws or clamps for tests upon individual strands of yarn shall be of the cylindrical or drum type, so arranged that the strands of yarn shall pass around not less than 180 deg. circumference before being clamped or fixed in the jaw. The length of the specimen shall be considered from center to center of drums. The minimum diameter of the cylinder or drum shall be ½ in.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, United States Rubber Co., 122 Adams St., Newark, N. J.

Jaw Dimensions.

6. The width of jaw in a direction perpendicular to the specimen shall in no case be less than 1 in. The depth of jaw in a direction length-wise of the specimen shall in no case be less than 1 in.

Backlash.

7. The dial pointer of textile testing machines shall be so arranged as to be easily adjustable to a zero reading for any weight of jaw or other fixture in the testing machine. The dial pointer shall be so counter-weighted as to prevent undue fluctuations in its position due to backlash, whatever the dial reading may be.

Machine Speed.

8. Textile testing machines shall be power driven or operated in such a manner as to produce a uniform and accurate movement of 12 in. per minute for the pulling jaw.

Calibration.

9. In calibrating textile testing machines, dead weights of accurate amounts shall be used, but these weights shall be applied at a speed of 12 in. per minute, corresponding to the standard jaw speed. The machine shall otherwise be arranged in an entirely similar manner to that used in testing fabric.

TENTATIVE SPECIFICATIONS
FOR
TOLERANCES AND TEST METHODS FOR COTTON YARNS, SINGLE AND PLIED[1]

Serial Designation: D 180 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision

ISSUED, 1923; REVISED, 1924, 1925.

1. These specifications cover the tolerances and methods of testing for cotton yarn, both single and plied. **Scope.**

TOLERANCES

I. STRENGTH

2. The average tensile strength of each case, bale, chain ball or beam warp of yarn, either single or plied, as found by test shall be not less than the specified strength. **Strength of Yarn.**

II. SIZE OR YARN NUMBER

3. The average size of each case, bale, chain ball or beam warp of yarn, in the singles, either carded or combed, as found by test shall not vary more than 3 per cent above or below the specified size. **Size of Yarn, Carded and Combed.**

Example.—No. 36 carded yarn specified; tolerance would allow 34.92 to 37.08.

III. TWIST AND DIRECTION OF TWIST

4. *Direction of Twist.*—In the case of yarn, the yarn has right-hand twist if, when it is held vertically, the spirals or twists are seen to incline upward in a right-hand direction, and has left-hand twist when the spirals or twists are seen to incline upward in a left-hand direction. **Definition.**

NOTE.—The definition as here given does not agree with the definition of "direction of twist" of cotton sewing thread as appearing in the Tentative Specification of Tolerances and Test Methods for Cotton Sewing Threads (D 204 - 24 T) which definition defines "direction of twist" in such a way that right-hand twist in the case of sewing thread corresponds to left-hand twist in the case of yarn, as defined herein. An attempt is being made to harmonize these two conceptions of "direction of twist."

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[1] **Criticisms of these Tentative Specifications are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, United States Rubber Co., 122 Adams St., Newark, N. J.**

Twist of Plied Yarns.

5. The average twist of each case, bale, chain ball or beam warp of plied yarns as found by test shall not vary more than 5 per cent above or below that specified.

Example.—Twist specified is 20 twists per inch; tolerance 19 to 21.

METHODS OF TESTING

IV. STRENGTH

Two test methods are given, the skein test for single yarn and the single strand test for single and plied yarns. A Preferred and Alternative Method for each test is given. The Alternative Method can be used where routine testing is done on a large scale. The Preferred Method should always be used in case of dispute.

Strength, Single Yarn,—Skein Preferred.

6. *Skein Test (Preferred Method).*—A standard skein (120 yd.) shall be broken after conditioning of tubes or bobbins selected for test for 12 hours or of skeins for at least 3 hours in an atmosphere of 65 per cent relative humidity, 70° F. (21° C.). An automatic power yarn tester of inclination balance type, the maximum capacity of which shall be determined in accordance with a table of machine specifications, shall be used. The drums of the testing machine shall consist of cylindrical spools not less than one inch in diameter and not less than one inch in width, so supported that at least one shall turn freely upon its axis. The speed of the pulling jaw shall be 12 in. per minute. Any yarn reel having a $1\frac{1}{2}$-yd. perimeter may be used in preparing the skeins. For filling-wound yarns or yarns on cones, where the yarn is drawn from the top, a speed of 100 to 300 r.p.m. of reel shall be used. For warp-wound yarns or yarn on parallel tubes where the yarn is drawn from the side, a speed of 20 to 30 r.p.m. of reel shall be used. On reels that have only one pigtail guide, the tension shall be applied by making one full wrap of the yarn around the guide. On reels using two or more guides, the yarn shall pass straight through the guides onto the reel, the angles of the guides supplying the necessary tension. Judgment must be used in regard to the amount of tension required on yarns having little or a large amount of twist. Three tests from each of four bobbins from every case of yarn shall be made.

7. *Strength Yarn on Beams (Skein Test for Yarn on Beams).*—Yarn received on beams shall be given the skein test and skeins shall be prepared in the following manner: Place the beam containing the yarn to be tested on two bearings sufficiently high from the floor so that the beam can be turned easily. Directly in front of the beam and at least 15 ft. from the beam, a small table shall be placed, the top of which will be approximately the same height as the top of the beam of yarn. On this table a standard yarn reel shall be placed directly in front of the beam. A small crank arm shall be attached

to one end of the beam shaft. Four ends of yarn, taken from the beam, shall be attached to the yarn reel in the regular manner, such as is done when testing yarn from bobbins. One operative shall turn the beam slowly in the proper direction so that the yarn will unwind, and the second operative shall turn the reel fast enough to take up the yarn as it comes off the beam. This operation shall be continued until 120 yd. or the desired length for testing has been taken from the beam. The four skeins shall then be taken from the reel and tested in the regular manner. If it should be necessary to test more ends, the same method of testing shall be followed.

NOTE.—In using these beams after these ends have been taken off, a set of spools containing the same kind of yarn as is on the beam can be placed in behind the beam on a small creel, to fill out the number of ends that have been used for testing. When the ends come up on the beams that have been used for testing, the auxiliary spools in back of the beams can be broken out.

Strength, Single Yarn, Single Strand Preferred.

8. *Single Strand Test* (*Preferred Method*).—Single strands shall be broken after conditioning the tubes or bobbins for 12 hours in an atmosphere of 65 per cent relative humidity, 70° F. (21° C.). A single strand tester of proper capacity with the jaws set 10 in. between grips and having a speed of pulling jaw of 12 in. per minute shall be used. The average of 4 breaks from each of 10 bobbins shall be the average strength.

Strength, Plied Yarns, Single Strand, Preferred.

9. *Plied Yarns* (*Preferred Method*).—Plied yarns, except standard tire cord, shall be subjected to the single strand break after conditioning for 12 hours on spools or tubes selected for test, in an atmosphere of 65 per cent relative humidity and 70° F. (21° C.). Standard tire cord shall be tested under dry conditions in accordance with the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[1] A single strand tester of proper capacity with the jaws set 10 in. between grips and having a speed of pulling jaw of 12 in. per minute shall be used. The average of 4 breaks from each of 10 spools or tubes shall be reported as the average strength.

Strength for All Yarns, Alternative Method.

10. *Skein and Single Strand* (*Alternate Method*).—Skeins or single strands of yarn either single or plied, prepared in accordance with Sections 6, 7, 8 and 9, shall be broken under natural humidity conditions at time of test. The results thus obtained shall be reduced to a common basis of standard moisture regain equal to 7 per cent of the bone-dry weight.

Moisture Regain Determination.

11. To determine moisture regain present in samples, the several skeins shall be weighed collectively, immediately after testing, under natural moisture conditions which obtain at the time of test. The skeins shall then be placed in the basket of an oven at a temperature

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[1] 1924 Book of A.S.T.M. Standards.

of 105 to 110° C. (221 to 230° F.) and dried to constant weight. The moisture regain is then computed as the percentage of the dry weight.

Correction to Standard Regain.

12. (*a*) The following formula shall then be applied, based on the assumption that the standard moisture regain of cotton yarns is 7 per cent of the dry weight, that the actual percentage regain is between the limits of 3 and 7 per cent of the dry weight and that for 1 per cent of moisture regain there is an increase of 6 per cent in the tensile strength of the yarn.

$$\text{Tensile strength corrected to standard moisture regain} = \frac{(\text{Tensile strength from machine reading}) \times 142}{100 + (6 \times \text{actual percentage regain.})}$$

Example.—With a skein of yarn broken under natural conditions showing a tensile strength of 120 lb. as read from the machine dial, and which by weighing before and after drying the skein was found to contain a moisture regain equal to 5 per cent of the bone-dry weight, the tensile strength corrected to a common basis of 7 per cent moisture regain would be:

$$\text{Corrected tensile strength} = \frac{120 \times 142}{100 + (6 \times 5)} = 131 \text{ lb.}$$

(*b*) Moisture regain tests shall be made periodically during the hours of testing as the natural humidity conditions are found to vary.

Strength Correction to Size.

13. The average tensile strength shall be corrected to the specified size as determined in accordance with Sections 14, 15 and 16, by the following formula:

$$\text{Corrected tensile strength} = \text{Actual average strength} \times \frac{\text{Actual average size}}{\text{Specified size}}$$

V. SIZE OR YARN NUMBER

Size of Single Yarns, Preferred Method.

14. The size of all standard skeins used in the skein strength test described in Section 6 shall be determined immediately after being broken. In case the single strand test is made, the standard skein shall be prepared for the size determination at the time of the break and the size determined immediately. The size of 60-yd. bundles prepared from beams as described in Section 7 shall be determined as soon as possible after conditioning and one test from each beam shall be reported as the average size. The balance to be used in this test shall be accurate to 0.25 per cent of the standard size of the yarn. When the balance does not indicate the size directly, the yarn number or size may be calculated from the formula:

$$\text{Yarn number or size} = \frac{\text{Length in yards of single yarn}}{\text{Weight in grains}} \times \frac{7000 \text{ (grains in 1 lb.)}}{840 \text{ (yards of No. 1 cotton yarn per pound)}}$$

Size of Plied Yarns, Preferred Method.

15. In determining the size of plied yarns, the skein shall be prepared in accordance with Table I, and the size shall be determined after conditioning of tubes or spools selected for test for 12 hours or of skeins for at least 3 hours, in an atmosphere of 65 per cent relative humidity, and 70° F. (21° C.). Any yarn reel having a $1\frac{1}{2}$-yd. perimeter may be used in preparing the skeins. For filling-wound yarns or yarn on cones, a speed of 100 to 300 r.p.m. of reel shall be used. For warp-wound yarns or yarn on parallel tubes, a speed of 20 to 30 r.p.m. of reel shall be used. On reels that have only one pigtail guide, the tension shall be applied by making one full wrap of the yarn around the guide. On reels using two or more guides, the yarn shall pass straight through the guides onto the reel, the angles of the guides supplying the necessary tension.

TABLE I.

EQUIVALENT SINGLES SIZE	YARDS FOR SIZE	CONVERSION FORMULA	NUMBER OF TESTS PER CASE OF YARN
20's and above	60	$\frac{\text{Size}}{2} = \text{Ply size}$	3 from each of 4 spools or tubes
3's to 20's	24	$\frac{\text{Size}}{5} = \text{Ply size}$	3 from each of 4 spools or tubes
Below 3's	12	$\frac{\text{Size}}{10} = \text{Ply size}$	3 from each of 4 spools or tubes

Size of All Yarns, Alternate Method.

16. All yarns used in the alternative method of testing for strength (Section 10) shall be sized under natural humidity conditions at the time of test. Plied yarns shall be prepared in skeins in accordance with Table I. The moisture regain shall then be determined as specified in Section 11, and results corrected to a common basis of standard moisture regain equal to 7 per cent of the bone-dry weight by means of the formula:

$$\text{Size corrected to standard moisture regain} = \frac{\text{Size} \times (100 + \text{actual percentage regain})}{107}$$

The average of these tests shall be the average size of case, bale, ball chain or beam warp of yarn.

VI. TWIST

Twist of Single Yarns.

17. No precision method for determining the twist of single yarns has been developed.

Twist of Plied Yarns.

18. The ply twist in yarns of 2 or more ply shall be determined on any standard twist counter with jaws set 10 in. apart. The strands shall be clamped in jaws under a definite tension by attaching weights.

The tension to be used shall be determined from the formula:

$$\text{Tension, in grams} = \frac{156 \text{ (Constant)}}{\text{Equivalent singles size}}$$

Example.—The weight for 23/11 would be as follows:

$$\frac{156}{2.09} = 76 \text{ g.}$$

The constant of 156 represents a tension which should be placed on yarn or cord to hold it sufficiently taut and still not remove any stretch.

Number of Tests. 19. Three twist tests on each of 4 packages of yarn from each case shall be made, and the average of these 12 tests shall be the average of the case.

TENTATIVE SPECIFICATIONS
FOR
TOLERANCES AND TEST METHODS FOR COTTON SEWING THREADS[1]

Serial Designation: D 204 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924; REVISED, 1925.

TOLERANCES

I. STRENGTH

Strength.

1. The average tensile strength of each case of sewing thread as found by test shall be not less than the specified strength.

II. YARDAGE

Yards per Pound.

2. When thread is sold by weight instead of on a guaranteed yardage unit, the yards per pound shall be indicated on each tube, spool or cone.

Variation.

3. The actual number of yards per pound or unit of sale, as found by test, shall not be more than 4 per cent less than the number of yards per pound or unit of sale specified.

III. TWIST

Definition.

4. *Direction of Twist.*—In the case of sewing thread, the thread as right-hand twist if, when it is held vertically, the spirals or twists are seen to incline upward in a left-hand direction, and has left-hand twist if the spirals or twists are seen to incline upward in a right-hand direction.

NOTE.—The definition as here given does not agree with the definition of "direction of twist" of yarn as appearing in the Standard Specifications for Tolerances and Test Methods for Electrical Cotton Yarns (D 203 - 25) and in the Tentative Specifications for Tolerances and Test Methods for Cotton Yarns, Single and Plied (D 180 - 25 T) which definitions define "direction of twist" in such a way that left-hand twist in the case of yarn corresponds to righ-hand twist in the case of sewing thread, as defined herein. An attempt is being made to harmonize these two conceptions of "direction of twist."

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, United States Rubber Co., 122 Adams St., Newark, N. J.

Balance. 5. The finished sewing thread shall be balanced in twist as nearly as possible and in no case shall a yard of thread when drawn from the side of a spool or package twist upon itself more than six times when brought together to form a loop.

IV. RUNNING QUALITIES

Running Qualities. 6. In addition to meeting the foregoing requirements, sewing thread shall be subjected to a sewing machine test under actual factory conditions. In the event of any thread apparently failing to operate properly, it shall be judged by comparison on any given machine under identical conditions, with thread from a lot known to have previously operated satisfactorily.

METHODS OF TESTING

V. STRENGTH

Strength. 7. All threads shall be tested by the single strand test method. A single strand tester of proper capacity with jaws set 10 in. between grips and having a speed of pulling jaw of 12 in. per minute shall be used. The average of ten breaks from each of five spools or packages shall be the average strength. Any test during which the thread breaks at the jaw due to pinching or cutting shall not be included in the average.

VI. YARDAGE

Yards per Pound. 8. In determining the yards per pound of sewing thread, the skeins shall be prepared in accordance with Table I. Any yarn reel having a 1½-yd. perimeter may be used in preparing the skeins. On reels that have only one pigtail guide, tension shall be applied by making one full wrap of the thread around the guide. On reels using two or more guides, the thread shall pass straight through the guides onto the reel, the angles of the guides supplying the necessary tension.

TABLE I.

EQUIVALENT SINGLE SIZE	YARDS FOR TEST	NUMBER OF TESTS PER CASE OF THREAD
Finer than 10's................	120	3 from each of 4 spools
10's and coarser...............	60	3 from each of 4 spools

The skeins shall then be weighed and the average weight of the 12 skeins shall be converted to yards per pound from the formula:

$$\text{Yards per lb.} = \frac{7000 \times (\text{number of yards in skein})}{\text{Average weight in grains of skein}}$$

VII. LENGTH OF PACKAGE

Length of Package. 9. (*a*) (*Preferred Method*).—The length of package of sewing thread shall be determined by multiplying the number of yards per pound, obtained as outlined in Section 8, by the net weight in pounds of the thread in package to be tested.

(*b*) (*Alternate Method*).—The length of package of sewing thread shall be determined by measuring the entire length over a reel having a $1\frac{1}{2}$-yd. perimeter, taking three full turns around the reel and delivering thread to a take-up drum. The yardage shall be noted on reel dial.

(*c*) (*Alternate Method*).—The length of package of sewing thread shall be determined by measuring the thread in the form of skeins prepared on a reel having a $1\frac{1}{2}$-yd. perimeter. In no case shall the yardage of these skeins exceed that specified in Table II. The total yardage of skeins thus prepared from a package constitutes its length.

TABLE II.

EQUIVALENT SINGLE SIZE	LENGTH OF SKEIN, YD.
Finer than 12's	240
6's to 12's, inclusive	120
Coarser than 6's	60

VIII. TWIST AND BALANCE

10. No precision method for determining the twist of single yarns has been developed. **Twist in Singles.**

11. Ply twist shall be determined on any standard twist counter with the jaws set 10 in. apart. The strands shall be clamped in jaws under a definite tension obtained by attaching weights. The tension to be used shall be determined from the formula: **Twist in Ply.**

$$\text{Tension, in grams} = \frac{156 \text{ (Constant)}}{\text{Equivalent singles size}}$$

Example.—The weight for 24/4 would be as follows:

$$\frac{156}{6} = 26 \text{ g.}$$

The constant of 156 represents a tension which should be placed on yarn or thread to hold it sufficiently taut and still not remove any stretch.

12. The "balance" of a sewing thread shall be determined by removing one yard of thread from a spool or package, holding it at each end and bringing these ends together, allowing the thread a chance to loop. **Balance.**

TENTATIVE SPECIFICATIONS
FOR
TOLERANCES FOR NUMBERED COTTON DUCK[1]

Serial Designation: D 230 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Tolerances. 1. Tolerances shall be the limit within which a textile must come in its specified characteristics in order that it shall constitute a good delivery on contract. They may be classified as the allowable limits of the quantitative characteristics of the fabric as defined in the specifications. The following tolerances are based upon the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[2]

I. WIDTH

Width. 2. The average width determined by measurement shall be as specified with the following tolerances:

WIDTH, IN.	TOLERANCE, IN. UNDER	TOLERANCE, IN. OVER
Up to and including 36	$\frac{1}{4}$	$\frac{1}{4}$
37 to 60, inclusive	$\frac{1}{4}$	$\frac{3}{8}$
61 to 80, inclusive	$\frac{3}{8}$	$\frac{5}{8}$
81 to 120, inclusive	$\frac{3}{8}$	$\frac{3}{4}$

II. WEIGHT

Weight. 3. The weight of the fabric determined by test shall be not more than 2.5 per cent over or under the specified weight.

III. THREADS PER INCH

Threads per Inch. 4. (*a*) The average count of warp ends per inch determined by test shall be not more than $1\frac{1}{2}$ ends over or under the specified count for fabrics counting not over 40 ends per inch and not more than 2 ends over or under for fabrics counting over 40 ends per inch.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, United States Rubber Co., 122 Adams St., Newark, N. J.

[2] 1924 Book of A.S.T.M. Standards.

(*b*) The average count of filling picks per inch determined by test shall be not more than 1 pick over or under the specified count for fabrics counting not over 25 picks per inch, and not more than 1½ picks over or under for fabrics counting from 25½ to 32 picks per inch, and not more than 2 picks over or under for fabrics counting over 32 picks per inch.

IV. STRENGTH

5. The average tensile strength of the warp and the average tensile strength of the filling shall be not less than the average specified tensile strength of each. **Strength.**

TENTATIVE SPECIFICATIONS
FOR
TOLERANCES AND TEST METHODS FOR KNIT GOODS[1]

Serial Designation: D 231 – 25 T

This is a Tentative Standard only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. These specifications are intended to be applicable to the examination of those classes of knit goods which later enter into a manufacturing process. Where a material requires special treatment, specific methods will be described as they are developed for that material and such special tests will have precedence over these general specifications.

TOLERANCES

I. WIDTH

Width. 2. The width as determined by test shall not be more than 3.5 per cent above or 3.5 per cent below the specified width.

II. WEIGHT

Weight. 3. The weight of the fabric as determined by test shall be not more than 5 per cent over or under the specified weight.

III. COUNT

Count. 4. The average count of wales per inch and courses per inch as determined by test shall be not more than 5 per cent over or under the specified count.

IV. TENSILE STRENGTH

Tensile Strength. 5. The average tensile strength of the fabric as determined by test shall be not less than the specified tensile strength.

METHODS OF TESTING

V. MOISTURE REGAIN

Moisture Regain. 6. (*a*) The moisture present shall be determined as follows: Portions of the samples which are taken for the determination of weight (as described in Section 10 (*a*)) weighing from 3 to 5 g. shall be placed in any suitable receptacle and the weight determined before and after drying in a ventilated oven at 105° C. The drying shall be continued until there is no change in weight, due care being taken to desiccate the receptacle and contents until cool, before weighing.

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[1] Criticism of these Tentative Specifications are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, United States Rubber Co., 122 Adams St., Newark, N. J.

(*b*) The loss in weight shall be considered moisture regain and the percentage estimated on the sample in bone-dry condition.

(*c*) Two determinations should be sufficient for ordinary purposes. Additional determinations may be required if desired.

VI. DETERMINATION OF GREASE

Determination of Grease.

7. (*a*) The amount of grease present shall be determined as follows: A representative sample which weighs about 5 g. taken from the samples prepared for strength tests shall be dried to constant weight in a ventilated oven at 105° C. It shall then be extracted with carbon tetrachloride in an apparatus as shown in Fig. 1, Section 8, of the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1] The extraction shall be continued for 8 hours and the extract dried to constant weight in a weighed flask at 105° C.

(*b*) The extract shall be considered grease and the percentage shall be reported on the dried sample.

(*c*) Two determinations of grease are usually sufficient. Additional determinations may be required if desired.

VII. PERCENTAGE OF COTTON AND WOOL

Percentage of Cotton and Wool.

8. (*a*) The amount of cotton and wool present shall be determined by the so-called "caustic-potash" method. This method shall be carried out as follows: The sample which has been dried for moisture determination shall be boiled thoroughly in 1-per-cent hydrochloric acid, but not long enough to disintegrate the fibers. This shall be repeated until no residue is found upon evaporating the solution on a watch glass. This removes sizing, dyestuff, mineral loading soluble in hydrochloric acid, etc. The residue shall be extracted successively with alcohol and carbon tetrachloride until all soluble material is removed. It shall be dried at 105° C. and weighed. This weight represents combined cotton and wool.

The dried residue shall be treated with a solution of 5-per-cent caustic potash and boiled gently for 15 minutes. It shall be poured into 500 cc. of cold water; permitted to stand until the fibers have settled and the supernatant liquid decanted. It shall be filtered through a Gooch crucible, washed with hot water and finally with

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[1] 1924 Book of A.S.T.M Standards.

10 cc. of alcohol. It shall then be dried and weighed. The loss in weight represents wool; the residue represents cotton, except that a correction shall be made for 3.5 per cent soluble cotton.

Example:

Preliminary Analysis		
Sample weight	5.000 g.	
Moisture	0.4370 g.	8.74 per cent
Sizing, grease, etc.	0.1640 g.	3.28 "
Fibers (by difference)	4.3990 g.	87.98 "
Analysis of Bone Dry Fibers		
Wool (loss to caustic potash)	2.3200 g.	52.74 "
Cotton (residue)	2.0790 g.	47.26 "
Cotton (corrected) (2.0790÷0.965)	2.1544 g.	48.97 "
Wool (corrected)	2.2446 g.	51.03 "
Air Dry Analysis		
Wool plus normal regain	2.5588 g.	50.99 "
Cotton plus normal regain	2.2945 g.	45.73 "
Sizing, grease, etc.	0.1640 g.	3.27 "

Assumptions	
Normal regain for wollen goods	14 per cent on dry weight
Normal regain for cotton goods	6.5 " " " "
Cotton soluble in caustic potash	3.5 " " " "

(*b*) Two determinations of percentage of cotton and percentage of wool are usually sufficient. Additional determinations may be required if necessary.

VIII. WIDTH

Width. 9. (*a*) The width of the roll or piece (either split or tubular) shall be determined at five different places uniformly distributed along the full length of the roll or piece. The goods shall be drawn across a table without tension when the width is measured.

(*b*) The average of the five measurements shall be the width.

IX. WEIGHT

Weight. 10. (*a*) Five samples which have been died out in staggered fashion across the roll or piece shall be weighed collectively. The die shall be square and 2 in. on a side. The material shall not be under tension as it is sampled.

(*b*) The samples shall be weighed under prevailing atmospheric conditions except in the settlement of disputes. In such cases, tests shall be made upon samples which have normal moisture content, obtained by exposure for at least 4 hours to an atmosphere of 65 per cent relative humidity at a temperature of 70° F. (21° C.); or upon agreement, the samples shall be weighed in the dry condition as described in Section 2 of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[1]

(*c*) The weight shall be reported as weight per square yard as calculated from the weight found.

X. THICKNESS

Thickness.

11. The thickness shall be measured by an automatic micrometer as described in Section 11 of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39).

XI. WALES AND COURSES

Wales and Courses.

12. The number of wales and courses per inch shall be determined by counting a space of not less than 2 in. in at least five different places in the roll or piece. The material shall be laid out on a table without tension while the wales and courses are being counted.

XII. TENSILE STRENGTH

Tensile Strength.

13. (*a*) The tensile strength shall be determined on an approved type of inclination balance breaking machine. The maximum capacity of the machine shall be such that the position of the pendulum shall not be greater than 45 deg. from the vertical when the break occurs. The minimum capacity shall not be less than 20 per cent of the above maximum capacity.

(*b*) The samples shall be tested under prevailing atmospheric conditions except in the settlement of disputes. In such cases, tests shall be made upon samples which have normal moisture content, obtained by exposure for at least 4 hours to an atmosphere of 65 per cent relative humidity at a temperature of 70° F. (21° C.); or upon agreement, the tests shall be carried out upon samples which have been bone-dried as described in Section 2 of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39).

(*c*) The 1 by 1 by 1-in. grab method shall be used as described in Section 19 (*d*) of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39), with the single exception that the distance between jaws shall be changed from 3 in. to 1 in.

(*d*) At least 20 test pieces 4 in. in width by 6 in. in length shall be cut, ten in the direction of the wales and ten in the direction of the courses.

(*e*) The tensile strength in each direction shall be the average of the ten tests.

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[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE METHODS OF TESTING GREASE WOOL AND ALLIED FIBERS FOR SCOURED CONTENT[1]

Serial Designation: D 232 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. These methods of test are intended to be applicable for testing grease wool and allied fibers (such as mohair, alpaca, camel hair, cashmere, vicuna) which contain grease, sand, dirt and vegetable matter, for the scoured content.

Form of Material. 2. The scoured content may be determined on the following forms of loose fiber in the grease:

(*a*) Wool (etc.) in the fleece;
(*b*) Necks, pieces, bellies;
(*c*) Pulled wool;
(*d*) Colonial scoured.

I. WOOL (ETC.) IN THE FLEECE

3. The tests to determine the scoured content of grease wool in the fleece shall be made on not less than 500 lb. but need not be on more than 1000 lb.

Weight. 4. (*a*) The wool shall be weighed in the original bags or bales at the time and place of making the test. This shall be considered the gross weight.

(*b*) The weight of the coverings and bindings and the string used for tying the fleeces, known as the tare, shall be determined.

(*c*) The difference between the gross weight and the tare shall be the "normal net weight" of the grease wool.

Sorting. 5. To retain the full commercial value of the wool after scouring it shall be sorted previous to scouring into an agreed number of main and off sorts.

Tags, Fribs, Clips. 6. The tags, fribs, clips and paint clips shall not be scoured, but shall be weighed in the grease and reported separately as a percentage of the normal net weight of the grease wool.

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, United States Rubber Co., 122 Adams St., Newark, N. J.

7. The main sorts and off sorts excepting tags, fribs, clips and paint clips, shall be scoured separately in a train of at least 4 bowls, or in 3 bowls and a rinse box. The first 3 bowls shall contain the detergents and the last bowl or the rinse box shall contain clean running water. **Scouring or Washing.**

8. The sorts shall be thoroughly dried in a heated mechanical dryer or on a heated table dryer and shall be bagged and weighed at once. **Drying.**

9. The total net weight of all the lots from the dryer, known as the "hot weight" of the scoured wool shall be determined. **Hot Weight.**

10. The difference between the normal net weight of the grease wool and the hot net weight of the scoured wool shall be determined as the "hot shrink" of the grease wool, when expressed as a percentage of the normal net weight of the grease wool by the following formula: **Hot Shrink.**

$$\text{Percentage Hot Shrink} = \frac{\begin{array}{c}\text{(Normal Net Weight of Grease Wool} - \\ \text{Hot Net Weight of Scoured Wool)} \times 100\end{array}}{\text{Normal Net Weight of Grease Wool}}$$

11. To determine the moisture in the hot net weight, three samples of not less than 1 lb. each shall be taken from the bags or bag representing about the beginning, the middle and the end of the run, immediately after the hot weight has been determined, and immediately placed in dry air-tight containers, the weights of which have been previously determined, and carefully weighed therein (check weight being made on containers); the net weight calculated, and the samples dried to constant weight in a ventilated drying oven maintained at a temperature of 230 to 240° F. (110 to 116° C.) as shown by two consecutive weighings not less than ten minutes apart without removing from the oven. **Moisture in Hot Net Weight.**

When two such weighings do not show a further loss of more than 0.1 per cent of the previous weighing the material is considered to be bone dry. **Bone Dry Weight.**

12. The difference between the average of the original (initial) weights of the samples, and the average of the bone dry weights of the same, shall be considered to be the moisture loss. **Actual Regain in Hot Weight.**

(*a*) When computed as a percentage of the initial weight it is the moisture content percentage.

(*b*) When computed as a percentage of bone dry weight it is the moisture regain percentage.

13. The standard condition of scoured wool shall be understood to be the condition in which it contains 12 per cent of its dry weight of moisture. **Standard Condition or Conditioned Weight.**

Standard Regain. 14. This 12 per cent shall be the standard regain of scoured wool.

Formula for Conditioned Weight. 15. The conditioned weight of the scoured wool shall be calculated from the following formula:

$$\text{Conditioned Weight} = \frac{\text{Hot Net Weight} \times 112}{100 + \text{Actual Regain}}$$

Conditioned Yield. 16. The true yield of the test lot shall be calculated from the following formula and shall be the actual yield of the entire lot.

$$\text{Yield, per cent} = \frac{\text{Conditioned Weight of Scoured Wool} \times 100}{\text{Normal Net Weight of Grease Wool}}$$

Conditioned Shrinkage. 17. If the loss in scouring is to be expressed as shrinkage, it shall be calculated from the following formula and shall be the actual shrinkage of the entire lot.

$$\text{Shrinkage, per cent} = \frac{(\text{Normal Net Weight of Grease Wool} - \text{Conditioned Weight of Scoured Wool}) \times 100}{\text{Normal Net Weight of Grease Wool}}$$

Thoroughly Scoured Wool. 18. Wool shall be considered thoroughly scoured when tests by the Soxhlet Extraction Apparatus do not show a loss of more than one per cent of the weight of the conditioned scoured wool before testing.

Scoured Wool Containing Grease. 19. Grease and solubles not to exceed a fixed percentage may be left in scoured wool by agreement, and tests to determine this percentage shall be made by the Soxhlet Extraction Apparatus.

II. NECKS, PIECES AND BELLIES

20. The tests to determine the scoured content of necks, pieces and bellies shall be made on not less than 500 lb. but need not be on more than 1000 lb. wool in the grease.

Weight. 21. (*a*) The wool shall be weighed in the original bags or bales at the time and place of making the tests. This shall be considered the gross weight.

(*b*) The weight of the coverings and bindings of the bales, known as the tare, shall be determined.

Normal Net Weight. (*c*) The difference between the gross weight and the tare shall be the "normal net weight" of the grease wool.

Scoured Content. 22. The test for scoured content shall be made as described for wool in the fleece in accordance with Sections 5 to 19, inclusive.

III. PULLED WOOL

23. The tests to determine the scoured content of pulled wool shall be made on not less than 500 lb. but need not be on more than 1000 lb. of wool in the grease.

24. (*a*) The wool shall be weighed in the original bags or bales at the time and place of making the tests. This shall be considered the gross weight. **Weight.**

(*b*) The weight of the coverings and bindings, known as the tare, shall be determined.

(*c*) The difference between the gross weight and the tare shall be the "normal net weight" of the grease wool.

25. The wool shall be scoured in a train of at least 4 bowls or in 3 bowls and a rinse box. The first 3 bowls shall contain the detergents and the last bowl or rinse box shall contain clean running water. **Scouring or Washing.**

26. The wool shall be thoroughly dried in a heated mechanical dryer or on a heated table dryer and shall be bagged and weighed at once. **Drying.**

27. The net weight of the wool from the dryer, known as the "hot weight" of the scoured wool shall be determined. **Hot Weight.**

28. The remainder of the test for determining the scoured content shall be made as described for wool in the fleece in accordance with Sections 10 to 19, inclusive. **Scoured Content.**

IV. COLONIAL SCOURED

29. The test to determine the scoured content of colonial scoured wool shall be made as described for pulled wool in accordance with Sections 23 to 28, inclusive.

TENTATIVE SPECIFICATIONS
FOR
SIEVES FOR TESTING PURPOSES[1]

Serial Designation: E 11 – 25 T

This is a Tentative Standard only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925

Sieves for precision testing have a variety of uses, including both dry screening and wet screening of material. The primary object is to separate materials according to the size of particle. Since particles are three dimensional and the openings of screens are areas having two dimensions only, the method of separation by means of screens is at best an approximation. In many instances, however, it is the best practical means at hand, and will give useful and reliable results if care is taken in securing representative samples, in the preparation of the sample for testing, in the details of procedure and in the method of reporting results. In the Appendix are indicated those points which must be considered in specifying sieving tests for any individual material.

An essential in each method, however, is a series of sieves of known characteristics. The important consideration in sieves is the size of opening and with the size of opening known it is possible to translate the results obtained in one series of sieves into terms of another series. The importance of the size of opening in the sieve series covered by these specifications is emphasized by designating the sieves in terms of opening in microns. This new scheme of designation should accordingly cause no confusion, giving as it does a direct indication of the size of opening. As such, it is not open to the objections to the various arbitrary number designations heretofore in use and it is also free from the objection to the designation by means of mesh in that it is independent of the size of wire.

With the size of opening known, the method of recording results is simplified. It is recommended that in reporting sieve analyses in graphic form the sizes of sieve opening be arranged as ordinates with the largest size at the top and that the percentages be plotted as abscissas.

Scope. 1. These specifications cover sieves for use in the usual cases of precision testing for the fineness of materials (classification of materials according to size). The sieves have been designated according to the size of openings in terms of microns, the corresponding United States Standard Sieve Series designation in arbitrary numbers being given for the convenience of those familiar with the latter scheme of designation.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. F. G. Breyer, Chairman of Sub-Committee, of Committee E-1, on Classification of Material According to Size, New Jersey Zinc Co., Palmerton, Pa.

I. PHYSICAL REQUIREMENTS

Cloth and Frame.

2. Wire cloth for standard sieves shall be woven (not twilled, except that the cloth of 44, 53 and 62-micron sieves may be twilled until further notice) from brass, bronze, or other suitable wire and mounted on the frames without distortion. To prevent the material being sieved from catching in the joint between the cloth and the frame, the joint shall be smoothly filled with solder, or so made that the material will not catch. The sieve frames should be circular, about 20 cm. (8 in.) in diameter and about 5 cm. (2 in.) or 2.5 cm. (1 in.) between the top of the frame and the cloth.

TABLE I.—REQUIREMENTS FOR SIEVE OPENINGS AND WIRE DIAMETERS WITH PERMISSIBLE VARIATIONS.

Designation	Corresponding U. S. Standard Sieve Series Number	Sieve Opening		Wire Diameter		Tolerance in Average Opening, per cent	Tolerance on Wire Diameter, per cent		Tolerance in Maximum Opening, per cent
		mm.	in.	mm.	in.		Under	Over	
	2	3	4	5	6	7	8	9	10
4760 micron......	4	4.76	0.187	1.27	0.050	±3	15	30	10
4000 "	5	4.00	0.157	1.12	0.044	±3	15	30	10
3360 "	6	3.36	0.132	1.02	0.040	±3	15	30	10
2830 "	7	2.83	0.111	0.92	0.036	±3	15	30	10
2380 "	8	2.38	0.0937	0.84	0.0331	±3	15	30	10
2000 "	10	2.00	0.0787	0.76	0.0299	±3	15	30	10
1680 "	12	1.68	0.0661	0.69	0.0272	±3	15	30	10
1410 "	14	1.41	0.0555	0.61	0.0240	±3	15	30	10
1190 "	16	1.19	0.0469	0.54	0.0213	±3	15	30	10
1000 "	18	1.00	0.0394	0.48	0.0189	±3	15	30	10
840 "	20	0.84	0.0331	0.42	0.0165	±5	15	30	25
710 "	25	0.71	0.0280	0.37	0.0146	±5	15	30	25
590 "	30	0.59	0.0232	0.33	0.0130	±5	15	30	25
500 "	35	0.50	0.0197	0.29	0.0114	±5	15	30	25
420 "	40	0.42	0.0165	0.25	0.0098	±5	15	30	25
350 "	45	0.35	0.0138	0.22	0.0087	±5	15	30	25
297 "	50	0.297	0.0117	0.188	0.0074	±6	15	35	40
250 "	60	0.250	0.0098	0.162	0.0064	±6	15	35	40
210 "	70	0.210	0.0083	0.140	0.0055	±6	15	35	40
177 "	80	0.177	0.0070	0.119	0.0047	±6	15	35	40
149 "	100	0.149	0.0059	0.102	0.0040	±6	15	35	40
125 "	120	0.125	0.0049	0.086	0.0034	±6	15	35	40
105 "	140	0.105	0.0041	0.074	0.0029	±8	15	35	60
88 "	170	0.088	0.0035	0.063	0.0025	±8	15	35	60
74 "	200	0.074	0.0029	0.053	0.0021	±8	15	35	60
62 "	230	0.062	0.0024	0.046	0.0018	±8	15	35	90
53 "	270	0.053	0.0021	0.041	0.0016	±8	15	35	90
44 "	325	0.044	0.0017	0.036	0.0014	±8	15	35	90

NOTE.—Screens having larger openings than those included in the table may be specified, but in specifying screens of larger openings the actual size of the opening (in millimeters or in inches) shall be stated. A tolerance of ±3 per cent on average openings and of 10 per cent in maximum openings shall be permitted.

NOTE.—The size specified above is for the regular standard testing sieve, but for special purposes other sizes may be used, as for example, the 3-in. sieve which is designed especially for use on laboratory balances as in the testing of pigments.

Permissible Variations.

3. The average opening between the adjacent warp and the adjacent shoot wires, taken separately, shall be that given in column

3 (or 4) of Table I, within the "Tolerance in Average Opening" given in column 7. The average diameter of the warp and of the shoot wires, taken separately, of the cloth of any given sieve shall be that given in column 5 (or 6) of Table I within the "Tolerance on Wire Diameter" given in columns 8 and 9. The maximum opening between adjacent parallel wires shall not exceed the nominal width of opening for that sieve by more than the "Tolerance in Maximum Opening" given in column 10 of Table I.

II. WORKMANSHIP

4. The sieves shall be free from imperfections in the sieve cloth or its mounting, as for example, punctured, loose or wavy cloth, imperfections in soldering, etc.

III. MARKING

5. The sieves shall bear a label upon which the manufacturer's nominal opening in metric units (preferably in microns) with the equivalent in inches, together with the size of wire should be given.

NOTE.—It is recommended that, until the new micron designation shall have been thoroughly established, in addition the corresponding United States Standard Sieve Series number be given.

IV. METHODS OF TESTING

Methods of Testing.

6. (*a*) To determine whether the cloth conforms to the requirements of these specifications, the wire diameter shall be measured in each direction by means of a micrometer-microscope or projection apparatus or other suitable apparatus of requisite precision, care being taken in all cases to avoid diffraction effects in measuring to the edge of a wire. Micrometer calipers and steel scales should not be used for this purpose. The mesh shall be determined by counting the number of openings for a distance of at least 6 in. in both directions of the sieve, using a scale, either glass or steel, and a microscope. The use of scales with special rulings for obtaining mesh by counting "interference bands" is recommended in cases where large numbers of any of the sieves with the smaller openings are to be measured. From the wire diameter and the mesh the average opening shall be computed.

NOTE.—No general rule can be given for the number of observations to be taken, except that they be sufficient to cause no change in the result if say 3 or 4 additional measurements are made.

(*b*) Measurements of large openings shall be made by means of the micrometer-microscope or the projection lantern or other suitable apparatus in the same way as wire diameters are measured.

NOTE.—In order to insure the securing of standard sieves it is possible to obtain from the manufacturer or dealer sieves which have been certified by the U. S. Bureau of Standards.

APPENDIX

LIST OF POINTS TO BE CONSIDERED IN DRAWING UP SPECIFICATIONS FOR A SIEVING TEST

I. APPARATUS.

1. Enumeration of sieves to be used.
2. Auxiliary apparatus required for collection and reduction of gross sample, sample containers, and apparatus other than the sieves used in making the tests.

II. SAMPLING.

1. Unit of sampling.
2. Method of collecting representative gross sample.
3. Size of gross sample.
4. Cautions to be taken in handling sample to prevent contamination or change.
5. Reduction of gross sample.

III. PREPARATION OF THE SAMPLE FOR TESTING.

1. Drying.
2. Mixing.
3. Special treatment, if any is necessary.
4. Selection of test portion.
5. Size of test portion.

IV. PROCEDURE.

A. Dry Screening:

1. Order in which the sieves are to be used.
2. Detailed description of method of shaking.
3. Detailed description of procedure other than method of shaking.
4. End point.

B. Wet Screening:

1. Liquid to be used.
2. Description of any preliminary separation by classification or other means.
3. Order in which screens are to be used.
4. Detailed description of procedure including amount and method of adding liquid, method of stirring or other agitation.
5. End point.

V. METHOD OF REPORTING RESULTS.

VI. ALLOWABLE VARIATIONS BETWEEN DUPLICATE TESTS ON THE SAME MATERIAL.

TENTATIVE METHODS OF TENSION TESTING OF METALLIC MATERIALS[1]

Serial Designation: E 8 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924; Revised, 1925.

These methods deal with the form and dimensions, the machining, and the testing of tension test specimens of metallic materials. Such matters as methods of taking samples from which to prepare test specimens are treated in the individual specifications for ferrous metals and for non-ferrous metals.

I. TEST SPECIMENS

1. In recommending the following specimens for use in tension tests it is not intended to exclude entirely the use of other test specimens for special materials or for special forms of material; for example, in tests of tubing and pipe the form of specimen recommended for plate, shape and flat material (Fig. 1) is not generally suitable for use, and a special form of specimen is specified. It is, however, recommended that wherever it is feasible the specimens here recommended be used.

Wire, Rods and Pipe.

2. Tension test specimens of wire are of the full-size diameter as drawn, and tension test specimens of rods of ductile metal are often of the full-size diameter as rolled. Tension test specimens of pipe and of tubing are frequently of full size as manufactured, in which case the ends should be plugged with metal plugs which do not extend within the gage marks.

Plates, Shapes and Flats.

3. The tension test specimen shown in Fig. 1 is recommended for plate, shape and flat material. The thickness of the specimen is that of the material tested.

Note.—When it is desired to use a specimen with a gage length of less than 8 in., the general proportions of Fig. 1 should be followed. Specimens with a gage length of 2 in. are occasionally used. When it is not convenient to machine specimens to the standard shape shown in Fig. 1, specimens may be used with edges machined parallel for the entire length of the specimen. If such specimens are machined with a tool cutting across the edges of the specimen it is important that the transverse tool marks be removed before the specimen is tested.

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. F. Moore, Chairman of Sub-Committee, of Committee E-1, on Mechanical Testing of Metallic Materials, University of Illinois, Urbana, Ill.

These methods are in effect a revision of the Methods for Tension Tests of the Standard Methods of Mechanical Testing of Metallic Materials. The standard methods, which were last published under the Serial Designation: E 1 - 18, have accordingly been withdrawn.

For thick plate material, it is usually preferable to use a machined specimen rather than to use a specimen the full thickness of the plate.

Up to the present time it has not been possible to fix a definite line between plate material and sheet material.

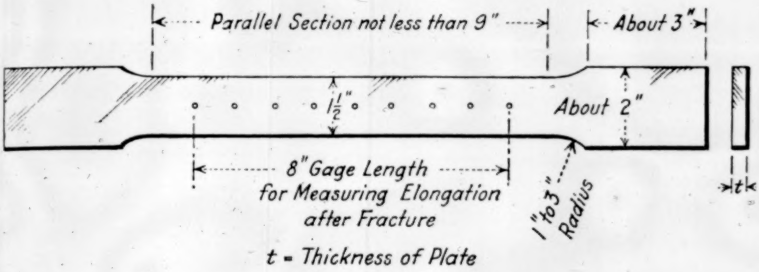

Parallel Section not less than 9"
About 3"
$1\frac{1}{2}$"
About 2"
8" Gage Length for Measuring Elongation after Fracture
1" to 3" Radius
t
t = Thickness of Plate

FIG. 1

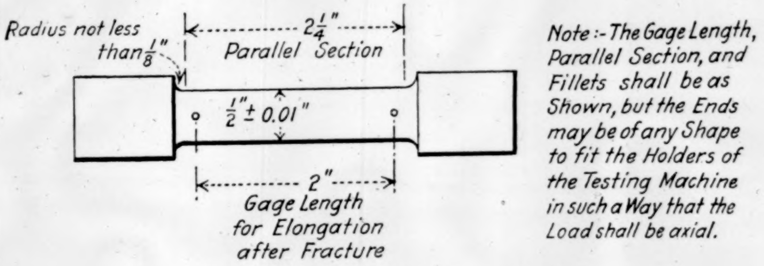
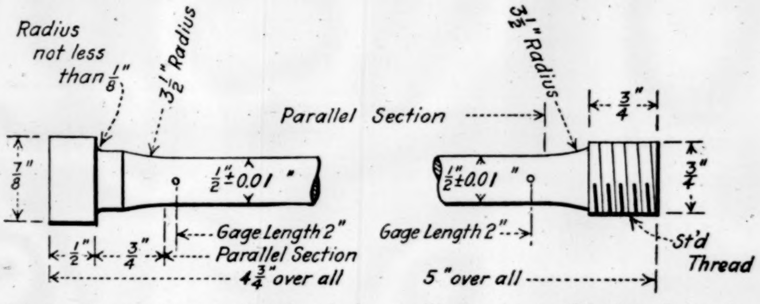

Radius not less than $\frac{1}{8}$"
$2\frac{1}{4}$"
Parallel Section
$\frac{1}{2}$" ± 0.01"
2"
Gage Length for Elongation after Fracture
Note:- The Gage Length, Parallel Section, and Fillets shall be as Shown, but the Ends may be of any Shape to fit the Holders of the Testing Machine in such a Way that the Load shall be axial.

FIG. 2


Radius not less than $\frac{1}{8}$"
$3\frac{1}{2}$" Radius
Parallel Section
$\frac{7}{8}$"
$\frac{1}{2}$" ± 0.01"
Gage Length 2"
$\frac{1}{2}$"
$\frac{3}{4}$"
Parallel Section
$4\frac{3}{4}$" over all
$3\frac{1}{2}$" Radius
$\frac{3}{4}$"
$\frac{1}{2}$" ± 0.01"
$\frac{3}{4}$"
Gage Length 2"
St'd Thread
5" over all

(a) Specimen with shouldered ends. (b) Specimen with threaded ends.

FIG. 3

4. The tension test specimen shown in Fig. 2 is recommended for general use in testing metals.[1] The gage length for measuring elongation after fracture is 2 in.

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[1] Attention is called to Section 7.

5. When it is necessary to cut specimens from material (other than plate, shape and flat material) which is of such size that the specimen shown in Fig. 2 cannot be used, it is recommended that a specimen with dimensions proportional to those shown in Fig. 2 be used and that the specimen be made as large as feasible. In any such small-size specimen it is especially important that the gage length for measuring elongation be four times the diameter of the specimen.

Measurement of Cross-Section.

6. In all tension tests of metals the actual dimensions of cross-section of the test specimens shall be measured with a micrometer reading to $\frac{1}{200}$ of the dimension measured, and the stresses shall be computed on the basis of the measured cross-section; it should never be assumed that the dimensions of the measured cross-section are identical with the nominal dimensions.

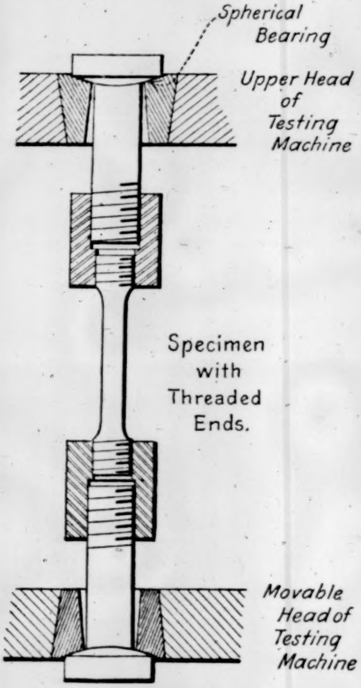
Spherical Bearing
Upper Head of Testing Machine
Specimen with Threaded Ends.
Movable Head of Testing Machine

FIG. 4

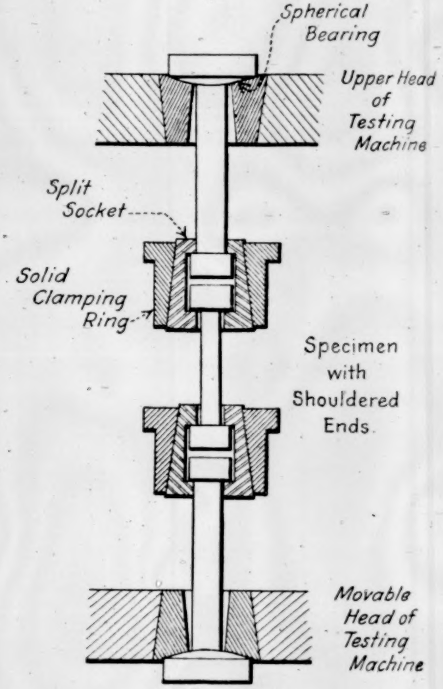
Spherical Bearing
Upper Head of Testing Machine
Split Socket
Solid Clamping Ring
Specimen with Shouldered Ends.
Movable Head of Testing Machine

FIG. 5

NOTE.—It will be noted that the tolerance for diameter of specimens shown in Fig. 2 permits the use of specimens 0.505 in. in diameter.

Ends.

7. The shape of the ends of the specimen shown in Fig. 2 are not specified above. For specimens of brittle metal it is desirable

that the diameter of the specimen be changed gradually along its length from the minimum section at the gage length to the diameter at the ends. Fig. 3 shows two forms of end of specimen which have given satisfactory results in tests of brittle metals.

8. Specimens shall be finished so that the surfaces are smooth and free from nicks and tool marks. All ragged edges shall be smoothed. **Finish.**

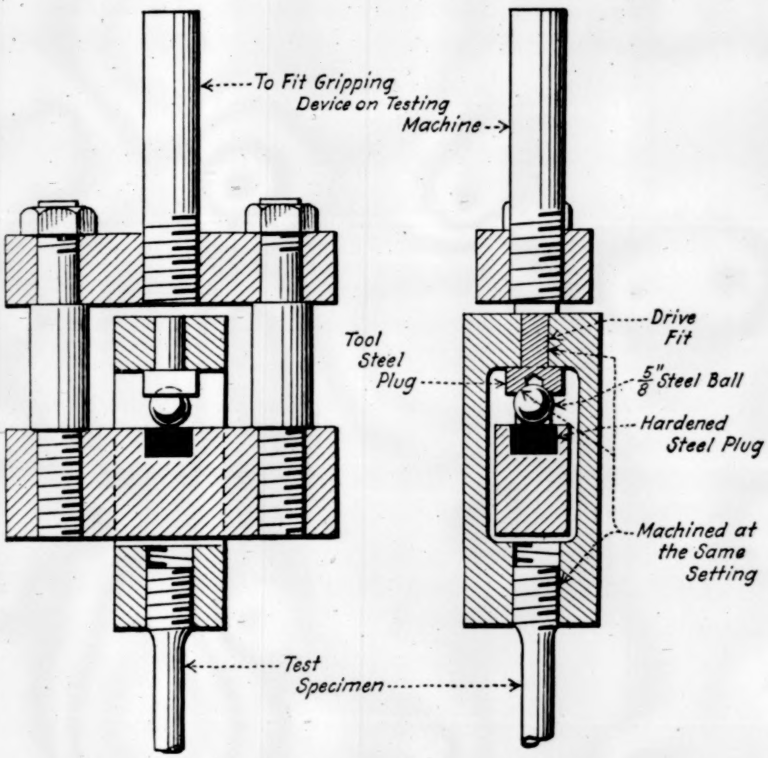
To Fit Gripping Device on Testing Machine
Tool Steel Plug
Drive Fit
$\frac{5}{8}$" Steel Ball
Hardened Steel Plug
Machined at the Same Setting
Test Specimen

FIG. 6

The gripping device is shown as used with threaded end specimens. A similar device fitted with split sockets would be used with shouldered specimens.

II. GRIPPING DEVICES FOR TENSION TEST SPECIMENS

9. For long bars of ductile metal and for the flat plate test specimen shown in Fig. 1, wedge grips, bearing directly on the end of the test specimen, generally furnish a satisfactory means of gripping the specimens. **Wedge Grips.**

10. For short specimens and for specimens of brittle material, wedge grips are not a satisfactory means of holding test specimens; it is necessary to use machined specimens and to use special means **Grips for Brittle Material.**

for insuring that the specimen, when under load, shall be as nearly as possible in pure axial tension. Fig. 4 shows a device for gripping tension specimens with threaded ends and Fig. 5 shows a device for gripping specimens with shouldered ends. Both these gripping devices are attached to the heads of the testing machine through spherical-seated bearings. The distance between spherical bearings should be as great as is feasible. For brittle materials, even the spherical-seated bearings shown in Figs. 4 and 5 are not always effective in avoiding bending stress on the specimen. The gripping device shown in Fig. 6 has proved satisfactory for testing such speci-

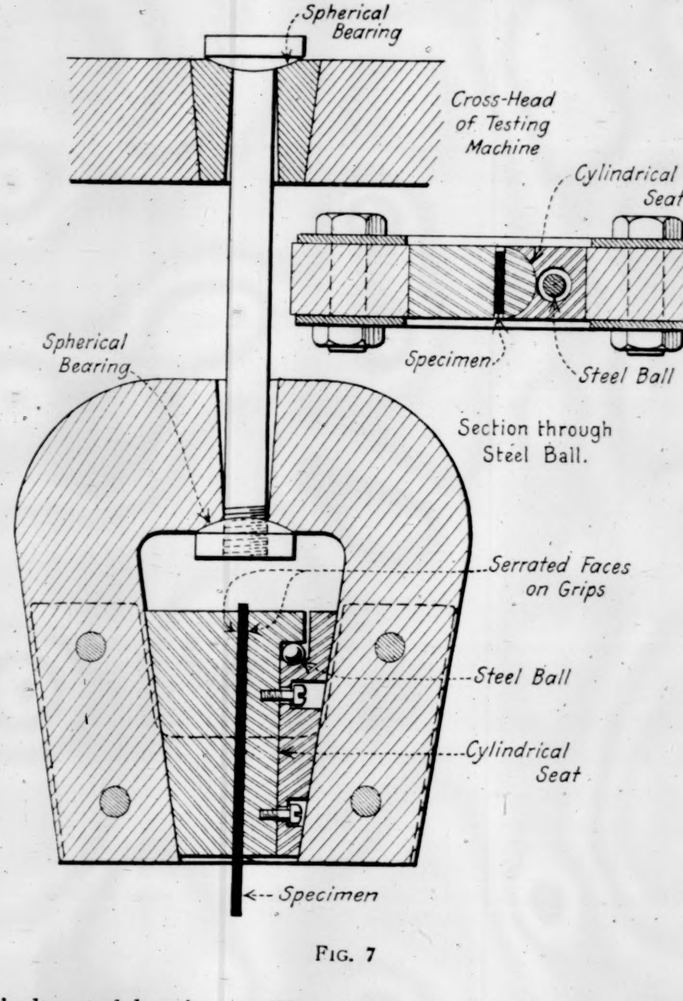
Spherical Bearing
Cross-Head of Testing Machine
Cylindrical Seat
Specimen
Steel Ball
Section through Steel Ball.
Spherical Bearing
Serrated Faces on Grips
Steel Ball
Cylindrical Seat
Specimen

FIG. 7

mens, and is an excellent gripping device for any machined tension test specimen.

11. For specimens cut from thin plate material and from sheet material, ordinary wedge grips are liable to cut the specimens near the edges, giving a tearing stress rather than an axial tension. The self-adjusting grips shown in Fig. 7 have proved satisfactory for tests of thin sheet copper and aluminum. **Grips for Sheet Material.**

12. For tests of specimens of wire, ordinary wedge grips are liable to cut the wire at the edges of the grips. This cutting has been found to be largely eliminated by the use of grips of the form shown in Fig. 8. **Grips for Wire.**

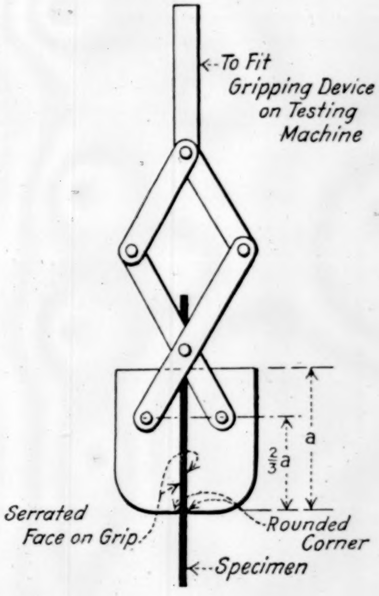

FIG. 8

13. In making tension tests, care shall be taken to see that the axis of the test specimen is located in the center line of the heads of the testing machine and that the liners used behind wedge grips are of proper thickness. From time to time the openings in the heads of the testing machine shall be lined up and the heads tested for parallelism. Deviations from correct alignment and from parallelism should not exceed 0.01 in. for any position of the movable head within the range of movement during test. The heads of the machine should be free from sidewise motion that is visible to the unaided eye as the machine is run at its fastest speed. **Alignment.**

III. SPEED OF OPERATION OF TESTING MACHINE

14. The pulling speed has a marked influence on the tensile properties shown by materials tested, an increase in speed increasing the values found for yield point and tensile strength. In testing specimens of metallic materials in gage lengths of 2 and 8 in., the speed of the machine, by which is meant the speed of the cross-head when the machine is running idle, shall conform to the following requirements:

The speed of head of the testing machine shall be such that the load can be accurately weighed, but in no case shall the values given in the following table be exceeded, which values represent the extreme of good laboratory practice for steel:[1]

Specified Minimum Tensile Strength of Material, lb. per sq. in.	Gage Length, in.	Maximum Cross-head Speed, in. per minute	
		Yield Point	Tensile Strength
80 000 or under	2 8	0.50 2.00	2.0 6.0
Over 80 000	2 8	0.25 0.50	1.0 2.0

15. In determining the elastic limit in accordance with Method I, Section 17, the cross-head speed for the 2-in. gage length shall not exceed 0.125 in. per minute.

16. In determining the elastic limit by Method II, Section 17, the cross-head speed shall not exceed 0.025 in. per inch of gage length per minute.

IV. THE DETERMINATION OF ELASTIC LIMIT, YIELD POINT, AND TENSILE STRENGTH

Elastic Limit.

17. The term "elastic limit" is defined as follows:

Elastic Limit.—The greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress.

The determination of the elastic limit as thus defined would logically involve the application and release of a succession of increasing loads on a test specimen until there was observed deformation after release of a load. This procedure is very slow and since for many materials experience does not indicate any appreciable difference between the elastic limit and the limit of proportionality of stress to strain (sometimes called the proportional limit), the determination of the stress at the limit of proportionality of stress to strain is regarded as an accurate determination of the elastic limit.

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[1] The speeds given here are probably too high for satisfactory use in testing non-ferrous metals.

It is obvious that the values obtained in tests for determining the elastic limit will depend on the delicacy of methods and instruments used. It becomes necessary, therefore, that in any test the method used in obtaining the elastic limit be clearly stated. The following methods are in common use for determining a value designated as the elastic limit.

Method I, proposed by Committee A-1 on Steel, and embodied in a number of specifications for steel. (For tension tests only.) The elastic limit shall be determined by an extensometer reading to 0.0002 in. The extensometer shall be attached to the specimen at the gage marks and not to the shoulders of the specimen nor to any part of the testing machine. When the specimen is in place and the extensometer attached, the testing machine shall be operated so as to increase the load on the specimen at a uniform rate. The observer shall watch the elongation of the specimen as shown by the extensometer and shall note, for this determination, the load at which the rate of elongation shows a sudden increase. The extensometer may then be removed from the specimen and the test continued to determine the tensile strength.

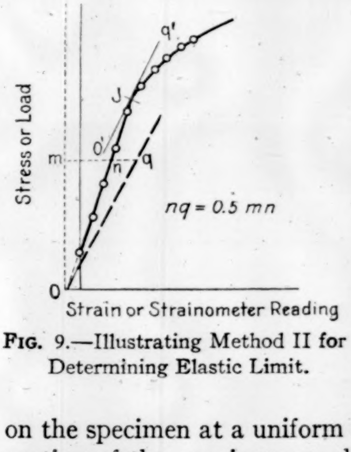

FIG. 9.—Illustrating Method II for Determining Elastic Limit.

Method II (proposed by the late J. B. Johnson) is a somewhat more delicate method for locating the elastic limit than is Method I. In Method II, the elastic limit shall be taken as that stress at which the rate of deformation is 50 per cent greater than the initial rate of deformation. A stress-strain diagram is necessary for determining the elastic limit by this method. It is illustrated in Fig. 9. The initial rate of deformation is given by the ratio $mn:Om$. $nq = 0.5\ mn$, $mq = 1.5\ mn$, and the slope of Oq represents a rate of deformation 50 per cent greater than the initial rate. $O'q'$ is drawn parallel to Oq and tangent to the stress-strain diagram. The point of tangency J

locates the elastic limit. In using Method II the same precautions should be observed in attaching the strainometer as for the determination of the elastic limit by Method I. Method II, in tension tests of metals, gives lower values for the elastic limit than does Method I. It is recommended that in using Method II for tension tests the strainometer be of such sensitiveness that it will indicate a change of strain of 0.0001 in. per inch of gage length (0.0001 mm. per millimeter of gage length).

Methods of High Precision.—When it is desired to determine the elastic limit with a high degree of precision, it is suggested that the experimenter devise his own procedure, and in reporting his results describe the procedure in detail, including a statement of the limits of sensitiveness of the apparatus used and of the procedure used in plotting the stress-strain diagram from which the determination of elastic limit is made. It is to be remembered that the accurate determination of the elastic limit requires the use of accurate and sensitive instruments and the use of accurate methods of plotting test data.

Yield Point.

18. The term "yield point" is defined as follows:

Yield Point.—The stress in a material at which there occurs a marked increase in strain without an increase in stress.

Two methods are in use for determining the yield point: (I) The "drop of the beam" method, and (II) the method by use of dividers:

Method I.—In Method I, load is applied to the specimen at a steady rate of increase and the operator keeps the beam in balance by running out the poise at a steady rate. At the yield point the increase of load stops (and for some metals there is an actual falling off of load), but the operator, running out the poise at a steady rate, runs it a trifle beyond the balance position, and the beam of the machine drops for a brief but appreciable interval of time. In a machine fitted with a self-indicating load-measuring device there is a sudden halt of the load-indicating pointer, corresponding to the drop of the beam. The load at the "halt" or the "drop" is recorded, and the corresponding stress is taken as the yield point. This method of determining the yield point requires only one man to conduct the test.

Method II.—In Method II an observer with a pair of dividers watches for visible elongation between two section marks on the specimens. When visible stretch is observed, the load at that instant is noted. and the stress corresponding to the load is taken as the yield point. When a strainometer[1] is used in place of dividers a deformation of 0.01 in. over a gage length of 2 in. shall be regarded as indicating the yield point.

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[1] The determination of yield point with a strainometer is sometimes made in tests on non-ferrous metals.

Some metals do not have a well-defined yield point.

Tensile Strength

19. The term "tensile strength" is defined as follows:

Tensile Strength.—The maximum tensile stress which a material is capable of developing.

NOTE.—In practice, it is considered to be the maximum stress carried by a specimen representing the material in a tension test to rupture, under definite prescribed conditions. Tensile strength is computed from the maximum load carried during a tension test and the original cross-sectional area of the specimen.

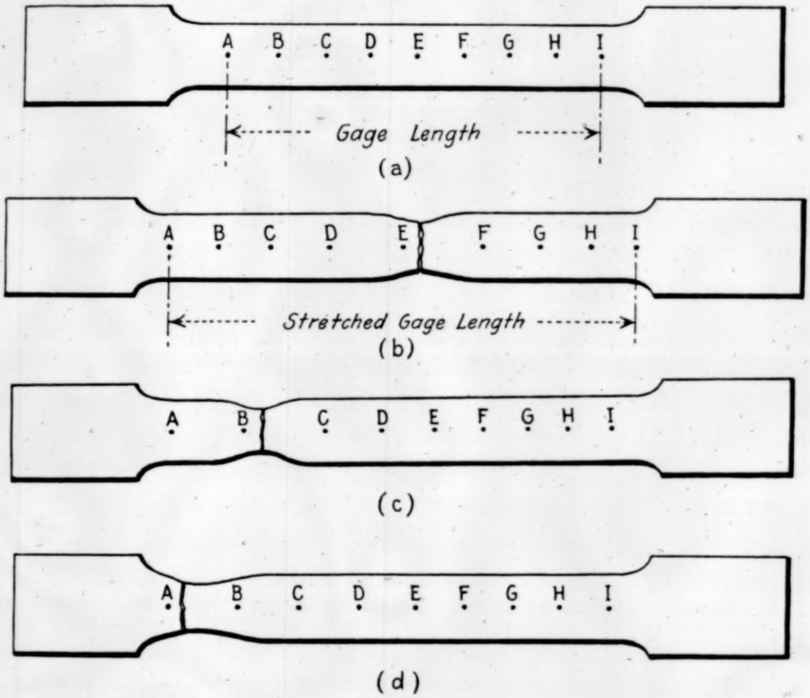


FIG. 10.

The conditions that must be prescribed in a tension test of metallic materials are the form of specimen, the method of gripping the specimen, and the rate of application of load. These are covered in foregoing sections. No further detailed discussion of methods of determining tensile strength is necessary, as there is involved merely the observing and the recording of the maximum load carried by the specimen during the test.

V. THE MEASUREMENT, AFTER FRACTURE, OF THE ELONGATION AND THE REDUCTION OF CROSS-SECTION OF TENSION TEST SPECIMENS

Elongation. 20. The measurement of the elongation after fracture of tension test specimens can be made with sufficient accuracy by means of a pair of dividers and a scale. The elongation should not be reported for any tension test specimen which breaks outside the middle third of the gage length.

NOTE.—If only a few specimens are furnished for testing it is recommended that in marking the gage length for measuring elongation after fracture it be divided into eight parts, and that if a specimen breaks inside the gage length but outside the middle third of the gage length the requirement in the foregoing paragraph be waived, and the elongation be measured and reported as follows:

(1) If the specimen breaks in the middle half of the gage length (between C and G, Figs. 10 (a) and (b)) the elongation is measured directly over the stretched gage length (Fig. 10 (b).

(2) If the specimen breaks between A and C (or between G and I) but nearer B than A (or nearer H than I), as shown in Fig. 10 (c) the elongation from A to C (or from G to I) is measured, and to this is added *twice* the measured elongation from C to F (or from D to G).

(3) If the specimen breaks within one-half a division of an end gage mark (see Fig. 10 (d)) the elongation from A to E (or from E to I) is measured and multiplied by 2.

The above method can be applied to specimens with any gage length, and to turned specimens as well as flat specimens.

This method is not applicable to brass specimens.

Reduction of Area. 21. The measurement of reduction of the dimensions of the cross-section of a tension test specimen may be made by the direct measurement with a micrometer of the smallest section of the fractured specimen. For round specimens this measurement can usually best be made by holding the broken pieces together in a vise or between centers and then measuring the average diameter of the smallest cross-section by means of a micrometer fitted with points so shaped that they will come in contact with the specimen at its smallest diameter.

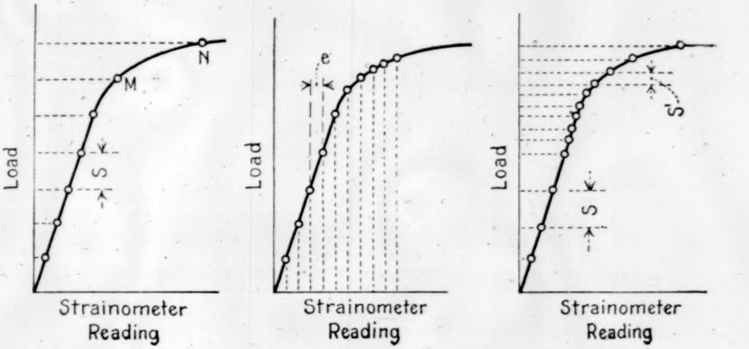
VI. PLOTTING STRESS-STRAIN DIAGRAMS

22. A stress-strain diagram is a diagram plotted with values of stress as ordinates and values of strain as abscissas.

NOTE.—The use of the term "stress-strain diagram" is frequently extended to cover diagrams plotted with values of applied load, or applied moment, as ordinates and with values of stretch, compression, deflection, or twist as abscissas.

Stress-strain diagrams are in some cases drawn directly by an autographic attachment to the testing machine. A more usual method of procedure consists in taking a series of load readings (from the balanced scale beam of the testing machine) with corresponding readings of the strain-indicating apparatus. The term "strainometer reading" will be used to denote the reading of the strain-measuring instrument. From these readings, or from values computed from them, there is plotted a diagram with stress-indicating values (load or stress) as ordinates, and strain-indicating values (elongation or strain) as abscissas. In planning such a test it is necessary to decide on the increment of load or the increment of reading of strainometer to be used between successive readings.

In Fig. 11 are shown three typical stress-strain diagrams. The material for all three tests is the same, and the test specimens are all of the same size, so that the diagrams should be the same. The diagram shown in Fig. 11 (*a*) is plotted from points determined by taking increments of load (*S*). In this diagram it is seen that owing to the shape of the curve, data for locating points between *M* and *N* are lacking. That particular portion of the curve (the "knee" of the curve) is the part for which it is especially desirable to locate several points on the diagram.



(*a*) With Equal Increments of Stress. (*b*) With Equal Increments of Strain. (*c*) With Two Values of Increments of Stress.

FIG. 11.—Illustrating Three Methods of Plotting Load-Deformation Curves.

In Fig. 11 (*b*) is shown a diagram plotted from points determined by taking increments of strainometer reading (*e*). It will be noted that for this diagram there are located several points near the knee of the curve, and the shape of the diagram in this important region is much more definitely determined than for the curve shown in Fig. 11 (*a*).

The custom of choosing increments of load rather than increments of strainometer reading is quite common in tests of materials and is followed because, in general, it is easier to compute increments of load than it is to compute increments of strainometer reading. An estimate of the load necessary to stress the specimen up to the knee of the curve is made and some fraction (frequently one-tenth) of this value is taken as an increment. The computation of the corresponding increment of strainometer reading is more complicated.

Sometimes the practice is followed of applying a few increments of load as determined above, and then applying load in much smaller increments (S') until the knee of the curve is passed. Fig. 11 (*c*) shows a diagram obtained in this manner. This method involves a marked increase in the number of readings necessary for a test and with unknown material there is always some danger that the knee of the curve will be reached before the use of small increments of load is begun.

The following method of choosing increments for a test is suggested: Estimate the load corresponding to the knee of the stress-strain diagram and choose a value for increment of load about one-tenth of this value. Apply this increment of load (S) once and note the corresponding change of reading for the strainometer (e). Then for the remainder of the test, use for the increment of strainometer reading a value which corresponds to some convenient interval on the scale of the strainometer, and which is approximately equal to (e).

TENTATIVE METHODS OF COMPRESSION TESTING OF METALLIC MATERIALS[1]

Serial Designation: E 9 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924; REVISED, 1925.

These methods deal with the form and dimensions, the machining, and the testing of compression test specimens of metallic materials. Such matters as methods of taking samples from which to prepare test specimens are treated in the individual specifications for ferrous metals and for non-ferrous metals.

I. TEST SPECIMENS

1. In recommending the following specimens for use in compression tests it is not intended to exclude entirely the use of other test specimens for special materials or for special forms of material. It is, however, recommended that wherever it is feasible the specimens here recommended be used.

2. It is recommended that standard compression specimens be in the form of circular cylinders. The ends of a specimen should be plane, as nearly as can be determined by the use of a straight edge and the unaided eye. In most cases this requirement necessitates the turning or the grinding of the ends of the specimen.

3. There are recognized three classes of compression specimens for metallic materials, designated as short specimens, medium-length specimens, and long specimens, respectively. Short specimens are used for compression tests of such metals as bearing metals, which in service are used in the form of a thin plate or shell to carry load perpendicular to the surface. Medium-length specimens are used for determining the general compressive strength properties of metallic materials. Long specimens are best adapted for determining the modulus of elasticity in compression of metallic materials. In reporting the results of a compression test, it is important that the dimensions of the test specimen be given.

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. F. Moore, Chairman of Sub-Committee, of Committee E-1, on Mechanical Testing of Metallic Materials, University of Illinois, Urbana, Ill.

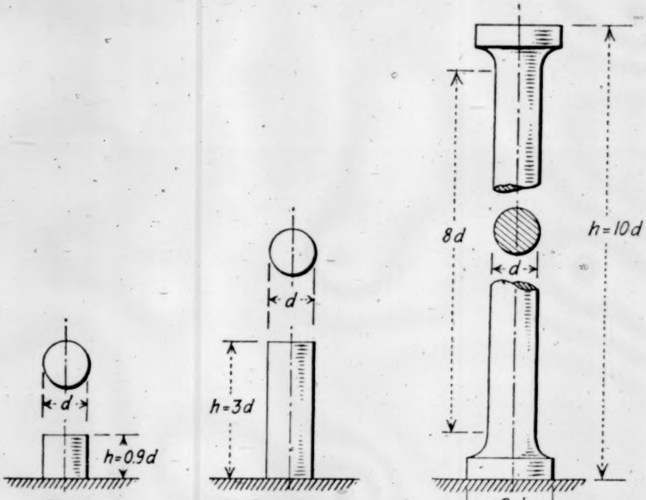
These methods are in effect a revision of the Methods for Compression Tests of the Standard Methods of Mechanical Testing of Metallic Materials. The standard methods, which were last published under the Serial Designation: E 1 - 18, have accordingly been withdrawn.

4. Fig. 1 shows the proportions of the three classes of compression specimens. Suggested dimensions for compression specimens for general use are:

Short specimens: $d = 1\frac{1}{8}$ in. ± 0.01 in. (area of cross-section is approximately 1 sq. in.), $h = 1$ in.;

Medium-length specimens: $d = 0.798$ in. ± 0.01 in., $h = 2\frac{3}{8}$ in., or
$d = 1$ in. ± 0.01 in., $h = 3$ in., or
$d = 1\frac{1}{8}$ in. ± 0.01 in., $h = 3\frac{3}{8}$ in.

Long specimens: $d = 1\frac{1}{4}$ in. ± 0.01 in., $h = 12\frac{1}{2}$ in.



Short Specimen. Medium-Length Specimen. Long Specimen.

FIG. 1.

5. In all compression tests of metallic materials, the actual dimensions of cross-section of the test specimens shall be measured with a micrometer reading to $\frac{1}{200}$ of the dimension measured, and stresses shall be computed on the basis of the measured cross-section. It should never be assumed that the dimensions of the cross-section of the specimen are identical with the nominal dimensions.

6. Specimens shall be finished so that the surfaces are smooth and free from nicks and tool marks. All ragged edges shall be smoothed.

II. BEARING BLOCKS FOR COMPRESSION TEST SPECIMENS

7. Both ends of a compression test specimen shall bear on a carefully machined plane surface. The bearing blocks shall either be made of hardened steel, or shall be faced with hardened steel.

8. One end of a compression test specimen shall bear on a spherical-seated compression block.[1] Fig. 2 shows a satisfactory arrangement of compression specimens and bearing blocks. It is desirable that the spherical-seated bearing block be at the upper end of the test specimen (for specimens tested with the axis vertical). It is important that the center of the spherical surface of the spherical-seated bearing block should be in the flat face which bears on the specimen, otherwise lack of parallelism between faces of the specimen sets up a bending moment in the specimen.[2]

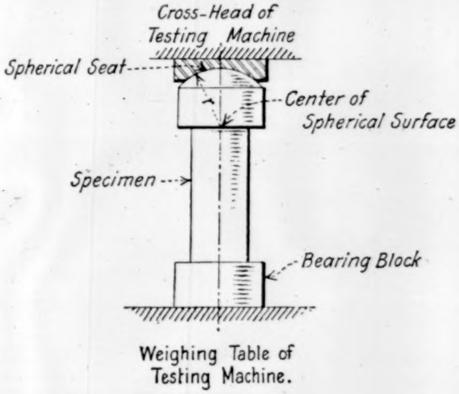

Cross-Head of Testing Machine
Spherical Seat
Center of Spherical Surface
Specimen
Bearing Block
Weighing Table of Testing Machine.

FIG. 2.

9. It should be borne in mind that the object of a spherical-seated bearing block is to give the specimen as even a distribution of initial load as is possible. Owing to friction, the spherical-seated bearing cannot be relied on to adjust itself to bending action which may occur during the test.

10. In making compression tests, care should be taken to preserve the alignment of the heads of the testing machine, and to center the specimen in the testing machine. From time to time the heads of the testing machine shall be tested for parallelism. Deviations from parallelism should not exceed 0.01 in. The movable head of the machine should be free from sidewise motion that is visible to the unaided eye as the machine is run at its fastest speed.

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[1] The device shown in Fig. 3 has been successfully used in compression tests of metallic specimens whose ends were machined with extreme care. It will be noted that this device does not involve a spherical seated compression block bearing directly on the test specimen. This device or one similar has been successfully used in the British National Physical Laboratory and in the laboratories of the U. S. Air Service at McCook Field, Dayton, Ohio.

[2] See Mont Schuyler, "Spherical Bearings," *Proceedings*, Am. Soc. Testing Mats., Vol. XIII, p. 1004 (1913).

III. SPEED OF OPERATION OF TESTING MACHINE

11. In making compression tests of specimens of metallic materials the speed of the machine, by which is meant the speed of the cross-head when the machine is running idle, shall not be greater than the values given in the following table:

LENGTH OF SPECIMEN, IN.	MAXIMUM CROSS-HEAD SPEED, IN. PER MINUTE
1.00	0.05
3.00 or greater	0.10

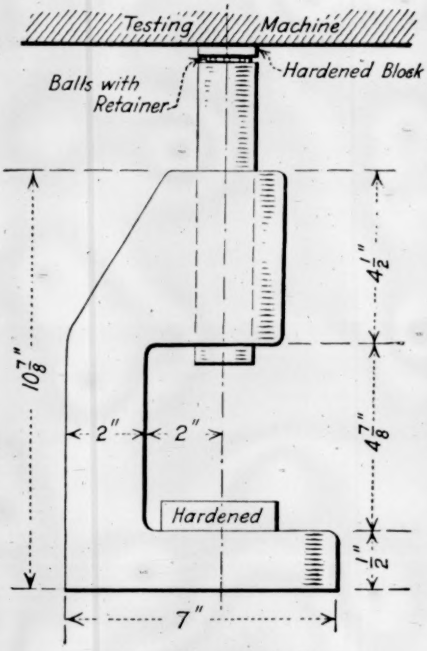
Testing Machine
Balls with Retainer
Hardened Block
$10\frac{7}{8}''$
$4\frac{1}{2}''$
$4\frac{7}{8}''$
$\frac{1}{2}''$
2″
2″
Hardened
7″

FIG. 3.—Compression Tool.

12. In making tests to determine the elastic limit in compression of metallic materials, the cross-head speed of the testing machine shall in no case exceed the allowable speed of head for the corresponding tension test of the material, as given in Sections 15 and 16 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 - 25 T) of the American Society for Testing Materials.[1]

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[1] See p. 854.

IV. THE DETERMINATION OF ELASTIC LIMIT, YIELD POINT, AND COMPRESSIVE STRENGTH

13. (*a*) The term "elastic limit" in compression is defined the same as in tension, as follows:

> *Elastic Limit.*—The greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress.

(*b*) The same general methods for determining elastic limit, as given in Section 17 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 - 25 T) of the American Society for Testing Materials,[1] shall be used. For compression tests, Method II or a still more precise method seems generally suitable.

14. (*a*). The term "yield point" in compression is defined the same as in tension, as follows:

> *Yield Point.*—The stress in a material at which there occurs a marked increase in strain without an increase in stress.

(*b*) The same methods for determining yield point as given in Section 18 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 - 25 T)[1] shall be used. The method by the use of dividers or by the use of a strainometer seems generally suitable for compression testing.

15. The term "compressive strength" is defined as follows:

> *Compressive Strength.*—The maximum compressive stress which a material is capable of developing.

NOTE.—In the case of a material which fails in compression by a shattering fracture the compressive strength has a very definite value. In the case of materials which do not fail in compression by a shattering fracture the value obtained for compressive strength is an arbitrary value depending upon the degree of distortion which is regarded as indicating complete failure of the metal.

V. PLOTTING STRESS-STRAIN DIAGRAMS

16. The discussion and directions given in Section 22 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 - 25 T)[1] apply to plotting stress-strain diagrams for compression tests.

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[1] See p. 854.

TENTATIVE METHODS OF BRINELL HARDNESS TESTING OF METALLIC MATERIALS[1]

Serial Designation: E 10 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924; Revised, 1925.

The Brinell test for determining the hardness of metallic materials consists in applying a known load to the surface of the material to be tested through a hardened steel ball of known diameter. The diameter (or depth) of the resulting permanent impression in the metal is measured. The Brinell hardness number is taken as the quotient of the applied load divided by the area of the surface of the impression, which is assumed to be spherical. If P is the applied load (measured in kilograms), D is the diameter of the steel ball (measured in millimeters) and d is the diameter of the impression (measured in millimeters), then

$$\text{B.H.N.} = \frac{P}{\frac{\pi D}{2}\left(D - \sqrt{D^2 - d^2}\right)}$$

in which B.H.N. is the Brinell hardness number in kilograms per square millimeter.

If the steel ball were not deformed under the applied load and if the impression were truly spherical, then the above formula would be a general one, and any combination of applied load and size of ball could be used. As the impression is not quite a spherical surface (since there must always be some deformation of the steel ball and some recovery of form of the metal in the impression), for a standard Brinell test the size and characteristics of the ball and the magnitude of the applied load must be standardized. (A standard ball 10 mm. in diameter and a load of 3000 kg. for hard metals and 500 kg. for soft metals is standard practice. See Sections 3 and 6.

In Table I is given the Brinell hardness number corresponding to various diameters of impression for both 500 and 3000 kg. load, making it unnecessary to compute for each test the value of the Brinell Hardness Number by the above formula.

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. F. Moore, Chairman of Sub-Committee, of Committee E-1, on Mechanical Testing of Metallic Materials, University of Illinois, Urbana, Ill.

These methods are in effect a revision of the Methods for Brinell Hardness Tests of the Standard Methods of Mechanical Testing of Metallic Materials. The standard methods, which were last published under the Serial Designation: E 1 – 18, have accordingly been withdrawn.

I. APPARATUS

1. The divisions on the micrometer scale of the microscope or other measuring device used for the measurement of the diameter of impression shall be such as to permit the direct measuring of the diameter to 0.1 mm. and the estimation of the diameter to 0.01 mm. **Micrometer Microscope.**

2. (*a*) The balls used in Brinell hardness testing shall be of hardened steel or other equally hard material. **Brinell Ball.**

(*b*) A ball to be suitable for use shall not show a permanent change in diameter greater than 0.0025 mm. (0.0001 in.) when pressed with a force of 3000 kg. against a piece of steel having a Brinell hardness number of 500 or greater.[1]

(*c*) If a ball is used in a test of a specimen which shows a Brinell hardness number greater than 500, the ball shall be remeasured after the test. Should the ball show a permanent change in diameter greater than that permitted in Paragraph (*b*), the ball shall be unsuitable for further use in a standard test.[1]

3. The standard ball for Brinell hardness testing shall be 10 mm. (0.3937 in.) in diameter with a deviation from this value of not more than 0.01 mm. (0.0004 in.) in any diameter, but the diameter of any ball measured at various points shall be constant within a tolerance of ±0.0025 mm. (0.0001 in.). **Size of Standard Ball.**

For testing very small specimens or very thin specimens it is sometimes necessary to make Brinell hardness tests with a ball less than 10 mm. in diameter. For such tests (which are not to be regarded as standard Brinell tests) the relation between applied load, P, measured in kilograms, and diameter of ball, D, measured in millimeters, should be

$$P = 3000\,\frac{D^2}{10^2} = 30\,D^2 \text{ for iron and steel}$$

$$\text{and } P = 500\,\frac{D^2}{10^2} = 5\,D^2 \text{ for brass, bronze and soft metals in general.}$$

For extremely soft metals the relation $P = D^2$ may be used.

Balls differing in size from the standard 10-mm. ball shall conform to the requirements for the material and the tolerance in size of diameter as specified for the standard ball.

II. TEST SPECIMENS

Specimens used in Brinell hardness testing vary greatly in form since it is frequently desirable to make the impression upon a part to be used in the finished product rather than upon a sample test specimen.

4. The thickness of the piece tested shall be such that no bulge or other marking showing the effect of the load appears on the side of **Thickness of Specimen.**

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[1] In testing softer metals, it is not, of course, necessary to have balls of this extreme hardness. However, a ball to be suitable for use should withstand pressure against the hardest material to be tested without showing a permanent change in diameter greater than 0.0025 mm.

TABLE I.—BRINELL HARDNESS NUMBERS.

Steel Ball, 10 mm. in Diameter, Pressures of 500 and 3000 kg.

Prepared by the Engineering Mechanics Section, Bureau of Standards.

The values given in this table for hardness numbers are merely solutions of the equation given in the introductory paragraphs. They do not imply that Brinell tests are feasible on materials of a hardness indicated by the highest values in the table.

Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number	
	500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load
2.00	158	945	2.50	100	601	3.00	69.1	415	3.50	50.3	302	4.00	38.1	229
2.01	156	936	2.51	99.4	597	3.01	68.6	412	3.51	50.0	300	4.01	37.9	228
2.02	154	926	2.52	98.6	592	3.02	68.2	409	3.52	49.7	298	4.02	37.7	226
2.03	153	917	2.53	97.8	587	3.03	67.7	406	3.53	49.4	297	4.03	37.5	225
2.04	151	908	2.54	97.1	582	3.04	67.3	404	3.54	49.2	295	4.04	37.3	224
2.05	150	899	2.55	96.3	578	3.05	66.8	401	3.55	48.9	293	4.05	37.1	223
2.06	148	890	2.56	95.5	573	3.06	66.4	398	3.56	48.6	292	4.06	37.0	222
2.07	147	882	2.57	94.8	569	3.07	65.9	395	3.57	48.3	290	4.07	36.8	221
2.08	146	873	2.58	94.0	564	3.08	65.5	393	3.58	48.0	288	4.08	36.6	219
2.09	144	865	2.59	93.3	560	3.09	65.0	390	3.59	47.7	286	4.09	36.4	218
2.10	143	856	2.60	92.6	555	3.10	64.6	388	3.60	47.5	285	4.10	36.2	217
2.11	141	848	2.61	91.8	551	3.11	64.2	385	3.61	47.2	283	4.11	36.0	216
2.12	140	840	2.62	91.1	547	3.12	63.8	383	3.62	46.9	282	4.12	35.8	215
2.13	139	832	2.63	90.4	543	3.13	63.3	380	3.63	46.7	280	4.13	35.7	214
2.14	137	824	2.64	89.7	538	3.14	62.9	378	3.64	46.4	278	4.14	35.5	213
2.15	136	817	2.65	89.0	534	3.15	62.5	375	3.65	46.1	277	4.15	35.3	212
2.16	135	809	2.66	88.4	530	3.16	62.1	373	3.66	45.9	275	4.16	35.1	211
2.17	134	802	2.67	87.7	526	3.17	61.7	370	3.67	45.6	274	4.17	34.9	210
2.18	132	794	2.68	87.0	522	3.18	61.3	368	3.68	45.4	272	4.18	34.8	209
2.19	131	787	2.69	86.4	518	3.19	60.9	366	3.69	45.1	271	4.19	34.6	208
2.20	130	780	2.70	85.7	514	3.20	60.5	363	3.70	44.9	269	4.20	34.4	207
2.21	129	772	2.71	85.1	510	3.21	60.1	361	3.71	44.6	268	4.21	34.2	205
2.22	128	765	2.72	84.4	507	3.22	59.8	359	3.72	44.4	266	4.22	34.1	204
2.23	126	758	2.73	83.8	503	3.23	59.4	356	3.73	44.1	265	4.23	33.9	203
2.24	125	752	2.74	83.2	499	3.24	59.0	354	3.74	43.9	263	4.24	33.7	202
2.25	124	745	2.75	82.6	495	3.25	58.6	352	3.75	43.6	262	4.25	33.6	201
2.26	123	738	2.76	81.9	492	3.26	58.3	350	3.76	43.4	260	4.26	33.4	200
2.27	122	732	2.77	81.3	488	3.27	57.9	347	3.77	43.1	259	4.27	33.2	199
2.28	121	725	2.78	80.8	485	3.28	57.5	345	3.78	42.9	257	4.28	33.1	198
2.29	120	719	2.79	80.2	481	3.29	57.2	343	3.79	42.7	256	4.29	32.9	198
2.30	119	712	2.80	79.6	477	3.30	56.8	341	3.80	42.4	255	4.30	32.8	197
2.31	118	706	2.81	79.0	474	3.31	56.5	339	3.81	42.2	253	4.31	32.6	196
2.32	117	700	2.82	78.4	471	3.32	56.1	337	3.82	42.0	252	4.32	32.4	195
2.33	116	694	2.83	77.9	467	3.33	55.8	335	3.83	41.7	250	4.33	32.3	194
2.34	115	688	2.84	77.3	464	3.34	55.4	333	3.84	41.5	249	4.34	32.1	193
2.35	114	682	2.85	76.8	461	3.35	55.1	331	3.85	41.3	248	4.35	32.0	192
2.36	113	676	2.86	76.2	457	3.36	54.8	329	3.86	41.1	246	4.36	31.8	191
2.37	112	670	2.87	75.7	454	3.37	54.4	326	3.87	40.9	245	4.37	31.7	190
2.38	111	665	2.88	75.1	451	3.38	54.1	325	3.88	40.6	244	4.38	31.5	189
2.39	110	659	2.89	74.6	448	3.39	53.8	323	3.89	40.4	242	4.39	31.4	188
2.40	109	653	2.90	74.1	444	3.40	53.4	321	3.90	40.2	241	4.40	31.2	187
2.41	108	648	2.91	73.6	441	3.41	53.1	319	3.91	40.0	240	4.41	31.1	186
2.42	107	643	2.92	73.0	438	3.42	52.8	317	3.92	39.8	239	4.42	30.9	185
2.43	106	637	2.93	72.5	435	3.43	52.5	315	3.93	39.6	237	4.43	30.8	185
2.44	105	632	2.94	72.0	432	3.44	52.2	313	3.94	39.4	236	4.44	30.6	184
2.45	104	627	2.95	71.5	429	3.45	51.8	311	3.95	39.1	235	4.45	30.5	183
2.46	104	621	2.96	71.0	426	3.46	51.5	309	3.96	38.9	234	4.46	30.3	182
2.47	103	616	2.97	70.5	423	3.47	51.2	307	3.97	38.7	232	4.47	30.2	181
2.48	102	611	2.98	70.1	420	3.48	50.9	306	3.98	38.5	231	4.48	30.0	180
2.49	101	606	2.99	69.6	417	3.49	50.6	304	3.99	38.3	230	4.49	29.9	179

TABLE I—(*Continued*).

Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number	
	500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load
4.50	29.8	179	5.00	23.8	143	5.50	19.3	116	6.00	15.9	95.5	6.50	13.3	79.6
4.51	29.6	178	5.01	23.7	142	5.51	19.2	115	6.01	15.9	95.1	6.51	13.2	79.3
4.52	29.5	177	5.02	23.6	141	5.52	19.2	115	6.02	15.8	94.8	6.52	13.2	79.0
4.53	29.3	176	5.03	23.5	141	5.53	19.1	114	6.03	15.7	94.4	6.53	13.1	78.7
4.54	29.2	175	5.04	23.4	140	5.54	19.0	114	6.04	15.7	94.1	6.54	13.1	78.4
4.55	29.1	174	5.05	23.3	140	5.55	18.9	114	6.05	15.6	93.7	6.55	13.0	78.2
4.56	28.9	174	5.06	23.2	139	5.56	18.9	113	6.06	15.6	93.4	6.56	13.0	77.9
4.57	28.8	173	5.07	23.1	138	5.57	18.8	113	6.07	15.5	93.0	6.57	12.9	77.6
4.58	28.7	172	5.08	23.0	138	5.58	18.7	112	6.08	15.4	92.7	6.58	12.9	77.3
4.59	28.5	171	5.09	22.9	137	5.59	18.6	112	6.09	15.4	92.3	6.59	12.8	77.1
4.60	28.4	170	5.10	22.8	137	5.60	18.6	111	6.10	15.3	92.0	6.60	12.8	76.8
4.61	28.3	170	5.11	22.7	136	5.61	18.5	111	6.11	15.3	91.7	6.61	12.8	76.5
4.62	28.1	169	5.12	22.6	135	5.62	18.4	110	6.12	15.2	91.3	6.62	12.7	76.2
4.63	28.0	168	5.13	22.5	135	5.63	18.3	110	6.13	15.2	91.0	6.63	12.7	76.0
4.64	27.9	167	5.14	22.4	134	5.64	18.3	110	6.14	15.1	90.6	6.64	12.6	75.7
4.65	27.8	167	5.15	22.3	134	5.65	18.2	109	6.15	15.1	90.3	6.65	12.6	75.4
4.66	27.6	166	5.16	22.2	133	5.66	18.1	109	6.16	15.0	90.0	6.66	12.5	75.2
4.67	27.5	165	5.17	22.1	133	5.67	18.1	108	6.17	14.9	89.6	6.67	12.5	74.9
4.68	27.4	164	5.18	22.0	132	5.68	18.0	108	6.18	14.9	89.3	6.68	12.4	74.7
4.69	27.3	164	5.19	21.9	132	5.69	17.9	107	6.19	14.8	89.0	6.69	12.4	74.4
4.70	27.1	163	5.20	21.8	131	5.70	17.8	107	6.20	14.8	88.7	6.70	12.4	74.1
4.71	27.0	162	5.21	21.7	130	5.71	17.8	107	6.21	14.7	88.3	6.71	12.3	73.9
4.72	26.9	161	5.22	21.6	130	5.72	17.7	106	6.22	14.7	88.0	6.72	12.3	73.6
4.73	26.8	161	5.23	21.6	129	5.73	17.6	106	6.23	14.6	87.7	6.73	12.2	73.4
4.74	26.6	160	5.24	21.5	129	5.74	17.6	105	6.24	14.6	87.4	6.74	12.2	73.1
4.75	26.5	159	5.25	21.4	128	5.75	17.5	105	6.25	14.5	87.1	6.75	12.1	72.8
4.76	26.4	158	5.26	21.3	128	5.76	17.4	105	6.26	14.5	86.7	6.76	12.1	72.6
4.77	26.3	158	5.27	21.2	127	5.77	17.4	104	6.27	14.4	86.4	6.77	12.1	72.3
4.78	26.2	157	5.28	21.1	127	5.78	17.3	104	6.28	14.4	86.1	6.78	12.0	72.1
4.79	26.1	156	5.29	21.0	126	5.79	17.2	103	6.29	14.3	85.8	6.79	12.0	71.8
4.80	25.9	156	5.30	20.9	126	5.80	17.2	103	6.30	14.2	85.5	6.80	11.9	71.6
4.81	25.8	155	5.31	20.9	125	5.81	17.1	103	6.31	14.2	85.2	6.81	11.9	71.3
4.82	25.7	154	5.32	20.8	125	5.82	17.0	102	6.32	14.1	84.9	6.82	11.8	71.1
4.83	25.6	154	5.33	20.7	124	5.83	17.0	102	6.33	14.1	84.6	6.83	11.8	70.8
4.84	25.5	153	5.34	20.6	124	5.84	16.9	101	6.34	14.0	84.3	6.84	11.8	70.6
4.85	25.4	152	5.35	20.5	123	5.85	16.8	101	6.35	14.0	84.0	6.85	11.7	70.4
4.86	25.3	152	5.36	20.4	123	5.86	16.8	101	6.36	13.9	83.7	6.86	11.7	70.1
4.87	25.1	151	5.37	20.3	122	5.87	16.7	100	6.37	13.9	83.4	6.87	11.6	69.9
4.88	25.0	150	5.38	20.3	122	5.88	16.7	99.9	6.38	13.8	83.1	6.88	11.6	69.6
4.89	24.9	150	5.39	20.2	121	5.89	16.6	99.5	6.39	13.8	82.8	6.89	11.6	69.4
4.90	24.8	149	5.40	20.1	121	5.90	16.5	99.2	6.40	13.7	82.5	6.90	11.5	69.2
4.91	24.7	148	5.41	20.0	120	5.91	16.5	98.8	6.41	13.7	82.2	6.91	11.5	68.9
4.92	24.6	148	5.42	19.9	120	5.92	16.4	98.4	6.42	13.6	81.9	6.92	11.4	68.7
4.93	24.5	147	5.43	19.9	119	5.93	16.3	98.0	6.43	13.6	81.6	6.93	11.4	68.4
4.94	24.4	146	5.44	19.8	119	5.94	16.3	97.7	6.44	13.5	81.3	6.94	11.4	68.2
4.95	24.3	146	5.45	19.7	118	5.95	16.2	97.3	6.45	13.5	81.0	6.95	11.3	68.0
4.96	24.2	145	5.46	19.6	118	5.96	16.2	96.9	6.46	13.4	80.7	6.96	11.3	67.7
4.97	24.1	144	5.47	19.5	117	5.97	16.1	96.6	6.47	13.4	80.4	6.97	11.3	67.5
4.98	24.0	144	5.48	19.5	117	5.98	16.0	96.2	6.48	13.4	80.1	6.98	11.2	67.3
4.99	23.9	143	5.49	19.4	116	5.99	16.0	95.9	6.49	13.3	79.8	6.99	11.2	67.0

the piece opposite the impression. In any event the thickness of the specimen shall be at least 10 times the depth of impression.

Finish of Specimen.

5. The surface on which the impression is to be made shall be filed, ground, machined, or polished with emery paper so that the edge of the impression shall be clearly enough defined to permit of the measurement of the diameter within 0.01 mm. (0.0004 in.).

III. APPLICATION AND MAGNITUDE OF LOAD

Magnitude.

6. The load in the standard Brinell test shall be 3000 kg. for iron and steel and 500 kg. for brass, bronze and soft metals generally.

NOTE.—For extremely soft metals a load of 100 kg. is sometimes used. If for any reason it is necessary to use loads differing from those specified above, the load used should be specifically stated in the test report, conveniently as a subscript of the hardness numeral.

Application.

7. (*a*) The load shall be applied steadily to the specimen without jerk.

(*b*) The load shall be applied for at least 10 seconds in the case of iron and steel and for at least 30 seconds in the case of other metals.[1]

(*c*) The center line of the applied load shall be far enough from any edge of the specimen so that when a straight-edge is applied to the edge after the load has been applied, the eye can detect no bulge in the edge due to the load and the resulting impression.

IV. MEASUREMENT OF IMPRESSION

8. In the standard Brinell hardness test the diameter of the impression shall be measured.

Measurement of Diameter of Impression.

9. When a micrometer microscope is used to measure the diameter of the impression the sharpness of definition of the edge of the impression can be increased by the use of a movable lamp for illuminating the specimen, placing the lamp so that the contrast of light and shade will bring first one edge of the impression, then the other into sharp definition. In testing very hard material, the sharpness of definition of the impression can be somewhat increased by the use of a ball lightly etched with nitric acid, or by the use of some pigment, such as Prussian blue, on the ball. In testing material in which there is considerable recovery of shape, the material may first be coated with a dull black pigment, such as drawing ink or a mixture of graphite and alcohol. The edge of the impression is rendered clear on a surface so coated.

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[1] **It was suggested in discussion that for magnesium and magnesium alloys the minimum time of application be 2 minutes. It was also suggested that for any given soft metal a definite time for holding the load be specified rather than a minimum time. See discussion of the Report of Committee E-1,** *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part 1, pp. 729 and 730 (1924).

BRINELL HARDNESS TESTS IN WHICH DEPTH OF IMPRESSION IS MEASURED IN PLACE OF DIAMETER

When it is necessary to make Brinell hardness tests very rapidly, as is sometimes the case when the Brinell test is used as a control test for the output of a heat-treating furnace, the depth of impression under load, or after the release of load, can be measured more expeditiously than the diameter of impression. The depth of impression is usually determined from the relative motion of the plunger carrying the ball and the specimen. This method of determining Brinell hardness is not to be regarded as a standard method, and when requirements of speed necessitate its use, the Brinell hardness number should not be determined from the geometrical relation of depth of impression to diameter, based on the assumption that the impression has a spherical surface. In such tests the relation between the Brinell hardness number and depth of impression may be determined with a fair degree of accuracy from tests on specimens of material of different degrees of hardness, several impressions being made on each specimen, and the depth under load, or the depth on release of load, and the diameter of impression after release of load being measured for each impression. By the use of a diagram or by interpolation, a table giving the Brinell hardness number for a given depth of impression can be prepared.

V. CALIBRATION OF BRINELL HARDNESS TESTING APPARATUS

10. A load-measuring device which is in common use for Brinell hardness testing machines involves the measurement of fluid pressure on a piston of known diameter, working in a cylinder with so perfect a fit that packing is not necessary. To this piston is attached the ball. Other load-measuring devices use combinations of weights and levers. The use of dead weights acting on a small piston is to be preferred to the ordinary Bourdon gage for measuring the intensity of fluid pressure.

11. The load-measuring device of a Brinell hardness testing machine may be calibrated by the use of dead weights, or by the use of dead weights and proving levers or by an elastic calibration bar or spring, in the manner described in the Standard Methods of Verification of Testing Machines (Serial Designation: E 4) of the American Society for Testing Materials.[1]

12. The load-measuring device of a Brinell hardness testing machine may be calibrated by making a series of impressions on specimens of different degrees of hardness, measuring the diameters of the impressions, making a second series of impressions by the use of any standardized testing machine and standard steel ball, and, using the same measuring device as for the first series of impressions, measuring the diameters of this second series of impressions. The error of the machine under calibration can be determined by the relative average

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[1] 1924 Book of A.S.T.M. Standards. See also J. L. Jones and C. H. Marshall, "A New Method for Calibrating Brinell Hardness Testing Machines," *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part II, p. 392 (1920).

values of the hardness numbers determined by its use and by the use of the standardized testing machine.

13. When the above companion-impression method of calibrating a Brinell hardness testing machine is used it is recommended that the specimens used be approximately 1 by 1 by 12 in. in size, and that specimens with as wide a range of hardness as is feasible be used. It is recommended that impressions made by the machine under calibration be spaced about 2.5 in. apart along the specimen, and that the impressions made by the standardized testing machine be spaced alternately along the length of the specimen with the impressions made by the machine under calibration.

14. If any determination of hardness, either for the machine under calibration or for the standard machine differs by more than 2 per cent from the mean of the four determinations made with that machine it is recommended that such determination be discarded. If two determinations differ more than 2 per cent from the mean the whole series should be discarded.

15. A Brinell hardness testing machine is acceptable for use over a loading range within which its load-measuring device is correct within 3 per cent.

16. The micrometer microscope or other device for measuring diameter of impression may be calibrated by using it to measure lengths on a standard steel or glass scale. The eye piece of a micrometer microscope should be adjusted so that throughout the range covered the error of reading does not exceed 0.01 mm.

TENTATIVE DEFINITIONS OF TERMS RELATING TO METHODS OF TESTING[1]

Serial Designation: E 6 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923; REVISED, 1924, 1925.

Stress.—The intensity (measured per unit area) of the internal distributed forces or components of force which resist a change in the form of a body. Stress is measured in force per unit area (pounds per square inch, kilograms per square millimeter, etc.).

NOTE.—In examining the definitions given in various authoritative text-books two definitions of the term "stress" were found. Text-books in physics and some European text-books in mechanics of materials gave a definition substantially as above. American text-books in engineering mechanics define stress as a *force*, measured in *pounds*. They call the intensity of force (pounds per square inch) "unit stress," "intensity of stress," or "fiber stress." This use of the term stress is illustrated by the "stress sheet" of the bridge engineer which gives *forces*, measured in pounds.

In view of the fact that even in engineering text-books the term "stress" is often used to denote intensity of force per unit of area, and in view of the fact that the physicists' definitions involve somewhat simpler terms, the physicists' definitions have been followed in these standards.

While it is important to have a clear definition for the term "stress" it is even more important to keep clearly in mind the *units* used in computations and test data for materials of construction. In fact, if the units are always given, there can be no misunderstanding as to the sense in which the term "stress" is used.

There are three kinds of stress: tensile, compressive, and shearing. Flexure involves the combination of tensile stress and compressive stress. Torsion involves shearing stress.

It is customary to compute stress on the basis of the original dimensions of the cross-section of the body.

Strain.—The change per unit of length in a linear dimension of a body, which change accompanies a stress. Strain is measured in inches per inch of length (millimeters per millimeter).

NOTE.—In American engineering text-books the term "strain" is used in the sense of total deformation and is measured in inches. Change of dimension per unit length is called "unit-strain," or "unit deformation." As in the consideration of the term "stress," the definitions given in text-books in physics have been followed.

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[1] Criticisms of these Tentative Definitions are solicited and should be directed to Mr. H. F. Moore, Chairman of Sub-Committee, of Committee E-1, on Mechanical Testing of Metallic Materials, University of Illinois, Urbana, Ill.

These definitions are in effect a revision of the definitions of terms appearing in the Standard Methods of Mechanical Testing of Metallic Materials. The standard methods, which were last published under the Serial Designation: E 1 - 18, have accordingly been withdrawn.

Under tensile stress or compressive stress, strain is measured along the dimension under consideration. Shearing strain is measured at right angles to the dimension under consideration. In torsion tests, which involve shearing stress, it is customary to measure the angle of twist, which may be translated into terms of strain.

Stress-Strain Diagram.—A diagram plotted with values of stress as ordinates and values of strain as abscissas.

NOTE.—The use of the term stress-strain diagram is frequently extended to cover diagrams plotted with values of applied load, or applied moment as ordinates, and with values of stretch, compression, deflection, or twist as abscissas.

Fig. 1 is an example of a stress-strain diagram.

Stress-strain diagrams are in some cases drawn directly by an autographic attachment to the testing machine. A more usual method of procedure consists in taking a series of load readings (from the balanced scale beam of the testing machine) with corresponding readings of the strain-indicating apparatus (extensometer, compressometer, deflectometer, or torsion indicator). The term "strainometer reading" will be used to denote the reading of any strain-measuring instrument. From these readings, or from values computed from them, there is plotted a diagram with stress-indicating values (load, flexural moment, twisting moment, or stress) as ordinates, and strain-indicating values (elongation, shortening, deflection, twist, or strain) as abscissas. In planning such a test it is necessary to decide on the increment of load or the increment of reading of strainometer to be used between successive readings.

In Fig. 2 are shown three typical stress-strain diagrams. The material for all three tests is the same, and the test specimens are all of the same size, so that the diagrams should be the same. The diagram shown in Fig. 2 (*a*) is plotted from points determined by taking increments of load (S). In this diagram it is seen that owing to the shape of the curve, data for locating points between M and N are lacking. That particular portion of the curve (the "knee" of the curve) is the part for which it is especially desirable to locate several points on the diagram.

In Fig. 2 (*b*) is shown a diagram plotted from points determined by taking increments of strainometer reading (e). It will be noted that for this diagram there are located several points near the knee of the curve, and the shape of the diagram in this important region is much more definitely determined than for the curve shown in Fig. 2 (*a*)

The custom of choosing increments of load rather than increments of strainometer reading is quite common in tests of materials and is followed because, in general, it is easier to compute increments of load than it is to compute increments of strainometer reading. An estimate of the load necessary to stress the specimen up to the knee of the curve is made and some fraction (frequently one-tenth) of this value is taken as an increment. The computation of the corresponding increment of strainometer reading is more complicated.

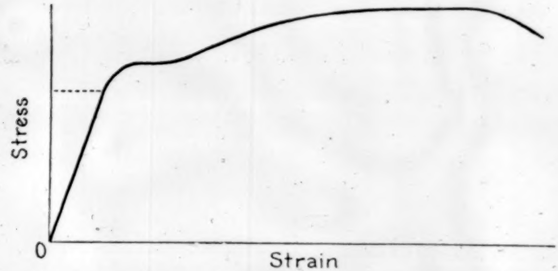
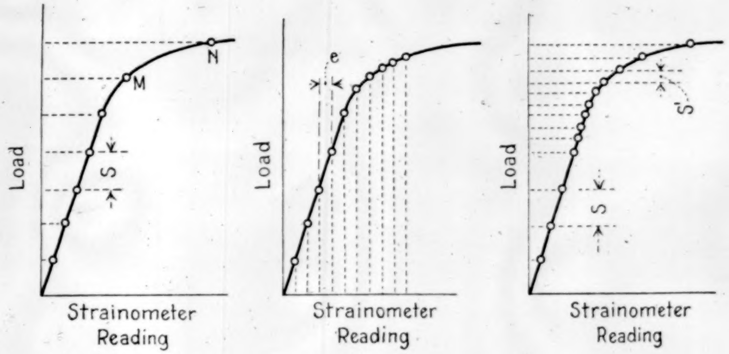


FIG. 1.—A Typical Stress-Strain Diagram.



(*a*) With Equal Increments of Stress.

(*b*) With Equal Increments of Strain.

(*c*) With Two Values of Increments of Stress.

FIG. 2.—Illustrating Three Methods of Plotting Load-Deformation Curves.

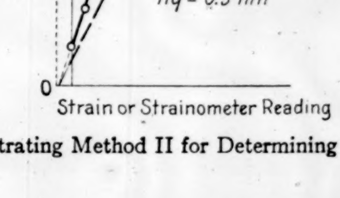


FIG. 3.—Illustrating Method II for Determining Elastic Limit.

Sometimes the practice is followed of applying a few increments of load as determined above, and then applying load in much smaller increments (S') until the knee of the curve is passed. Fig. 2 (c) shows a diagram obtained in this manner. This method involves a marked increase in the number of readings necessary for a test and with unknown material there is always some danger that the knee of the curve will be reached before the use of small increments of load is begun.

The following method of choosing increments for a test is suggested: Estimate the load corresponding to the knee of the stress-strain diagram and choose a value for increment of load about one-tenth of this value. Apply this increment of load (S) once and note the corresponding change of reading for the strainometer (e). Then for the remainder of the test, use for the increment of strainometer reading a value which corresponds to some convenient interval on the scale of the strainometer, and which is approximately equal to (e).

Elastic Limit.—The greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress.

NOTE.—In certain specifications of the Society the term "elastic limit" is used to designate a value obtained by a test method which does not involve the release of stress during a test, but which does involve the determination of a limiting load at which there occurs an appreciable change in rate of strain with respect to stress. This limit is frequently given the name "proportional limit" (or "proportional elastic limit"), and the following definition may be given:

Proportional Limit.—The greatest stress which a material is capable of developing without a deviation from the law of proportionality of stress to strain (Hooke's Law).

It is a matter of experience with many materials, especially with many metallic materials, that using ordinary methods of testing, the values found for elastic limit by means of observations of permanent deformation (set) after release of stress do not differ widely from the values found for proportional limit. Since the determination of proportional limit is much more readily made than is the determination of elastic limit it is customary to accept the proportional limit as equivalent to the elastic limit for such materials.

Using any known method, the value determined either for proportional limit or for elastic limit is somewhat arbitrary; the value depends on various test conditions, among them precision of apparatus, the scales to which stress-strain diagrams may be plotted, and the tolerance allowed before it is judged that there exists an appreciable deviation from the law of proportionality of stress to strain or an appreciable permanent deformation (set) upon release of stress.

In connection with this note on elastic limit and proportional limit, it seems proper to consider the action of a material under steadily increasing stress, and to discuss briefly five stages which may be distinguished between the first application of stress and fracture of the metal. Not all materials show all five stages, and for a material the end of one stage and the beginning of the next stage are not always marked.

In the first stage (OA, Fig. 4), with the use of measuring instruments of ordinary sensitiveness, deviations from the law of proportionality of stress to strain (Hooke's Law) will not be detected, nor will there be detected set after release of stress.

In the second stage (*AB*, Fig. 4) as stress increases there is detected an appreciable and growing deviation from the law of proportionality of stress to strain. This is usually accompanied by an appreciable and growing permanent deformation (set) after release of stress, although for some materials (*e.g.*, rubber) measurable permanent deformation may not occur after release of stress exceeding the proportional limit. Values for elastic limit and for proportional limit are found in this second stage.

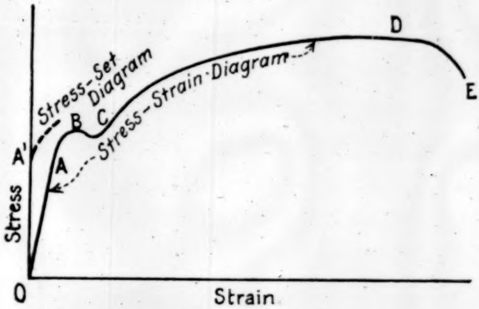


FIG. 4.

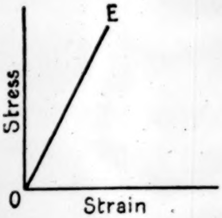


FIG. 5.

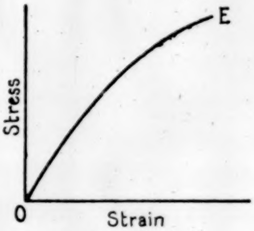


FIG. 6.

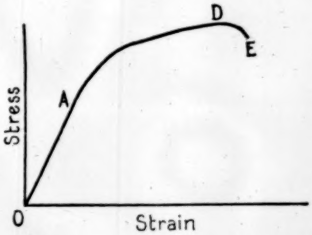


FIG. 7.

The third stage (*BC*, Fig. 4) is characteristic of ductile metals. In this stage, strain increases without any increase of stress, and in some cases increase of strain is accompanied by an actual decrease of stress. The yield point is found at the beginning of this stage (at *B*, Fig. 4).

In the fourth stage (*CD*, Fig. 4) stress and strain both increase up to the maximum stress carried (*D*, Fig. 4, which gives the tensile strength in a tension test of a material).

In the fifth stage (*DE*, Fig. 4) strain increases while stress diminishes from the maximum value (*D*, Fig. 4) to fracture (*E*, Fig. 4). In considering each of these stages it is important to note that stress is computed on the basis of original cross-section of specimen.

Many materials, especially brittle materials, do not exhibit all the above-mentioned stages. Some (*e.g.*, glass and fused quartz) show only the first stage. Fig. 5 gives a representative stress-stain diagram for such materials. Some materials (*e.g.*, cast iron) show only the second stage, and Fig. 6 shows a typical stress-strain diagram for such materials. Some materials (*e.g.*, medium-hard steel) do not show the third stage, and the second stage merges into the fourth stage. Fig. 7 shows a typical stress-strain diagram for such materials. The fifth stage is absent in many materials; it is, in fact, characteristic of ductile metals only.

The following methods of test are, strictly speaking, methods of determining proportional limit with various degrees of precision. However, the values determined by these methods may, for certain materials, be regarded as satisfactory and reliable values for the elastic limit, and they are therefore so used in certain specifications of the Society.

Method I. (For tension tests only, proposed by Committee A-1 on Steel, and embodied in a number of specifications for steel.) The elastic limit shall be determined by an extensometer reading to 0.0002 in. The extensometer shall be attached to the specimen at the gage marks and not to the shoulders of the specimen nor to any part of the testing machine. When the specimen is in place and the extensometer attached, the testing machine shall be operated so as to increase the load on the specimen at a uniform rate. The observer shall watch the elongation of the specimen as shown by the extensometer and shall note, for this determination, the load at which the rate of elongation shows a sudden increase. The extensometer may then be removed from the specimen and the test continued to determine the tensile strength.

Method II (proposed by the late J. B. Johnson) is a somewhat more delicate method for locating the elastic limit than is Method I. In Method II, the elastic limit shall be taken as that stress at which the rate of deformation is 50 per cent greater than the initial rate of deformation. A stress-strain diagram is necessary for determining the elastic limit by Method II. Fig. 3 illustrates the use of Method II. The initial rate of deformation is given by the ratio $mn : Om$. $nq = 0.5\ mn$, $mq = 1.5\ mn$, and the slope of Oq represents a rate of deformation 50 per cent greater than the initial rate. $O'q'$ is drawn parallel to Oq and tangent to the stress-strain diagram. The point of tangency J locates the elastic limit. In using Method II the same precautions should be observed in attaching the strainometer as for the determination of the elastic limit by Method I Method II, if used for tension tests of metals, gives lower values for the elastic limit than does Method I. It is recommended that in using Method II for tension tests the strainometer be of such sensitiveness that it will indicate a change of strain of 0.0001 in. per inch of gage length (0.0001 mm. per millimeter of gage length) and that a corresponding degree of sensitiveness be required for strainometers used for compression tests, for flexure tests, and for torsion tests.

When it is desired to determine the elastic limit with a high degree of precision, it is suggested that the experimenter devise his own procedure, and in reporting his results describe the procedure in detail, including a statement of the limits of sensitiveness of the apparatus used and of the procedure used in plotting the stress-strain diagram from which the determination of elastic limit is made. It is to be remembered that the accurate determination of the elastic limit requires the use of accurate and sensitive instruments and the use of accurate methods of plotting test data.

Yield Point.—The stress in a material at which there occurs a marked increase in strain without an increase in stress.

NOTE.—Some metals do not show a clearly defined yield point.

Two methods are in use for determining the yield point: (I) The "drop of the beam" method, and (II) the method by use of dividers. In Method I, load is applied to the specimen at a steady rate of increase and the operator keeps the beam in balance by running out the poise at a steady rate. At the yield point the increase of load stops, but the operator, running out the poise at a steady rate, runs it a trifle beyond the balance position, and the beam of the machine drops for a brief but appreciable interval of time. In a machine fitted with a self-indicating load-measuring device there is a sudden halt of the load-indicating pointer, corresponding to the drop of the beam. The load at the "halt" or the "drop" is recorded, and the corresponding stress is taken as the yield point. This method of determining the yield point requires only one man to conduct a test.

In Method II, an observer with a pair of dividers watches for visible elongation between two section marks on the specimens. When visible stretch is observed, the load at that instant is noted, and the stress corresponding to the load is taken as the yield point. When a strainometer[1] is used in place of dividers a deformation of 0.01 in. over a gage length of 2 in. shall be regarded as indicating the yield point.

Tensile Strength.—The maximum tensile stress which a material is capable of developing.

NOTE.—In practice, it is considered to be the maximum stress carried by a specimen representing the material in a tension test to rupture, under definite prescribed conditions. Tensile strength is computed from the maximum load carried during a tension test and the original cross-sectional area of the specimen.

Compressive Strength.—The maximum compressive stress which a material is capable of developing.

NOTE.—In the case of a material which fails in compression by a shattering fracture the compressive strength has a very definite value. In the case of materials which do not fail in compression by a shattering fracture the value obtained for compressive strength is an arbitrary value depending upon the degree of distortion which is regarded as indicating complete failure of the metal.

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[1] The determintion of yield point with a strainometer is sometimes made in tests on non-ferrous metals.

Modulus of Elasticity.—The ratio, within the elastic limit of a material, of stress to corresponding strain.

NOTE.—As there are three kinds of stress, so are there three moduli of elasticity for any material: the modulus in tension, the modulus in compression, and the modulus in shear. The value of the modulus of elasticity in tension is nearly the same, for most metals, as the value of the modulus of elasticity in compression. The value of the modulus of elasticity in shear is smaller than the value of the modulus of elasticity in tension. The modulus of elasticity is expressed in pounds per square inch (kilograms per square millimeter).

TENTATIVE DEFINITIONS OF TERMS RELATING TO SPECIFIC GRAVITY[1]

Serial Designation: E 12 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

1. *Absolute Specific Gravity* (of solids and liquids).—The ratio of the weight referred to vacuum of a given volume of the material at a stated temperature to the weight referred to vacuum of an equal volume of gas-free distilled water[2] at a stated temperature. It shall be stated thus:

(*a*) When the temperature of the material and that of the water are the *same:*

Absolute Specific Gravity x° C., ...,

where x is the temperature of the material and the water.

(*b*) When the temperature of the material and that of the water are *not the same:*

Absolute Specific Gravity $\frac{x^\circ \text{ C.},}{y^\circ \text{ C.}}$ ....,

where x is the temperature of the material and y the temperature of the water.

2. *Specific Gravity* (of solids and liquids).—The ratio of the weight in air of a given volume of the material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperature of the material and that of the water are the *same:*

Specific Gravity x° C., ...,

where x is the temperature of the material and the water.

(*b*) When the temperature of the material and that of the water are *not the same:*

Specific Gravity $\frac{x^\circ \text{ C.},}{y^\circ \text{ C},}$ ....,

where x is the temperature of the material and y is the temperature of the water.

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[1] Criticisms of these Tentative Definitions are solicted and should be directed to Mr. F. M. Farmer, Chairman of Sub-Committee, of Committee E-8, on Definition of Specific Gravity, Electrical Testing Laboratories, Eightieth Street and East End Ave., New York City.

[2] Distilled water boiled vigorously in vacuum.

3. *Apparent Specific Gravity* (of solids).—The ratio of the weight in air of a given volume of the impermeable portion of a permeable material (that is, the solid matter including its impermeable pores or voids) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperature of the material and that of the water are the *same:*

Apparent Specific Gravity $x°$ C., ...,

where x is the temperature of the material and the water.

(*b*) When the temperature of the material and that of the water are *not the same:*

Apparent Specific Gravity $\frac{x° \text{ C.,}}{y° \text{ C.}}$ ....,

where x is the temperature of the material and y is the temperature of the water.

NOTE.—In scientific circles, specific gravity and density determinations made in air (that is, uncorrected to vacuum) are frequently distinguished by the adjective "apparent." Thus, the specific gravity defined by definition No. 2 would be designated as "apparent specific gravity" and that defined by definition No. 1 as "specific gravity." But in industry, the terminology is more generally in accordance with that given in these definitions.

4. *Bulk Specific Gravity* (of solids).—The ratio of the weight in air of a given volume of a permeable material (including both permeable and impermeable voids normal to the material) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperature of the material and that of the water are the *same:*

Bulk Specific Gravity $x°$ C., ...,

where x is the temperature of the material and the water.

(*b*) When the temperature of the material and that of the water are *not the same:*

Bulk Specific Gravity $\frac{x° \text{ C.,}}{y° \text{ C.}}$ ....,

where x is the temperature of the material and y is the temperature of the water.

NOTES.—The terms "permeable" and "impermeable" cannot be rigidly defined for general application. The exact meaning in a particular application is the conventional one inferred by the procedure specified for determining the specific gravity of the material in question.

It is obvious that in the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed whenever possible.

TENTATIVE DEFINITION OF THE TERM SCREEN (SIEVE)[1]

Serial Designation: E 13 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Screen (*Sieve*).—A plate or sheet or a woven cloth, with regularly spaced apertures of uniform size, mounted on a suitable frame or holder, for use in separating materials according to size.

NOTE.—The shape and spacing of apertures, size of wires or threads, thickness of plate or sheet, allowable variations and similar properties, should be taken care of in specifications.

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[1] Criticisms of this Tentative Definition are solicited and should be directed to Mr. L. R. Ferguson, Chairman of the Sub-Committee, of Committee E-8, on Definitions of Sieve and Screen, 342 Madison Ave., New York City.

TENTATIVE RECOMMENDED PRACTICE
FOR
THERMAL ANALYSIS OF STEEL[1]

Serial Designation: E 14-25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

I. APPARATUS

Apparatus. 1. The apparatus required for thermal analysis consists of a furnace for heating the specimen, a thermocouple, a potentiometer or galvanometer for measuring small changes in e. m. f. and a stop-watch or chronometer.

Furnace. 2. An electric-resistance furnace provided with a means of accurate and smooth temperature control should be used. A furnace of the Rosenhain type or U. S. Bureau of Standards modification, is recommended. A good furnace is essential for accurate results. A neutral or reducing atmosphere or partial vacuum is desirable to protect the specimen from excessive oxidation.

Thermocouple. 3. The thermocouple may be of any approved type which has been calibrated either by the Bureau of Standards or against a secondary standard or by the melting point of pure metals (Tin 231.9° C., Lead 327.5° C., Zinc 419.4° C., Aluminum 658.7° C., Silver 960.5° C., Copper 1083° C.). A charge of at least 60 g. of metal should be used.[2] Base metal couples should not be used for temperatures exceeding 900° C.

4. For recording data, a sensitive galvanometer or potentiometer and stop-watch or a recording galvanometer and a chronograph should be used.

II. SPECIMENS

Specimens. 5. The size of specimen must be governed by the kind of furnace and the sensitivity of the instruments used. About ½ by ½ by 1 in. is satisfactory for most work.

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[1] Criticisms of this Recommended Practice are solicited and should be directed to Mr. G. F. Comstock, Secretary of Committee E-4 on Metallography, 967 Harrison Ave., Niagara Falls, N. Y.

[2] The couple should be inserted in the specimen, insulated from it with mica or asbestos.

The specimen should be normalized before making a thermal analysis.

NOTE—This requirements applies to routine work only. For special researches exception may be made.

III. PROCEDURE

Rate of Heating and Cooling.

6. The specimen should be heated and cooled at a uniform rate of from 5 to 10° C. (9 to 18° F.) per minute.

Maximum Temperature.

7. The specimen should be heated to a temperature of 100° C. above the upper critical point and allowed to remain there fifteen minutes before cooling is begun.

Interpretation of Curves.

8. Data may be plotted as desired. The inverse rate method is the simplest and most satisfactory for all general purposes and the most generally used.

In fixing the critical points, the temperature should be read at the cusp, or maximum point, of the inflection of the curve.

TENTATIVE REVISIONS
OF
A.S.T.M. STANDARDS

Tentative revisions of A.S.T.M. Standards are printed for one or more years with a view of eliciting criticism, of which the committee concerned will take due cognizance before recommending final action.

Members of the Society and others are invited to direct written criticisms of any of these Tentative Revisions to the officer of the appropriate committee, whose name and address appear in the footnote in connection with each revision.

TENTATIVE REVISIONS OF A.S.T.M. STANDARDS

A: FERROUS METALS

STANDARD SPECIFICATIONS FOR CARBON-STEEL AND ALLOY-STEEL FORGINGS

(Serial Designation: A 18 – 21)[1]

STANDARD SPECIFICATIONS FOR QUENCHED-AND-TEMPERED CARBON-STEEL AXLES, SHAFTS AND OTHER FORGINGS FOR LOCOMOTIVES AND CARS

(Serial Designation: A 19 – 21)[1]

STANDARD SPECIFICATIONS FOR CARBON-STEEL FORGINGS FOR LOCOMOTIVES

(Serial Designation: A 20 – 21)[1]

STANDARD SPECIFICATIONS FOR QUENCHED-AND-TEMPERED ALLOY-STEEL AXLES, SHAFTS AND OTHER FORGINGS FOR LOCOMOTIVES AND CARS

(Serial Designation: A 63 – 21)[1]

In Specifications A 18 – 21 change Section 4, and in Specifications A 20 – 21 change Section 5 from their present form: namely,

> "The manufacturer and the purchaser shall agree upon forgings on which a prolongation for test purposes shall be provided."

and in Specifications A 19 – 21 and A 63 – 21 change Section 4 from its present form: namely,

> "For test purposes, a prolongation shall be left on each forging, unless otherwise specified by the purchaser."

to read as follows:

> "Unless otherwise specified, for test purposes at least 20 per cent of the forgings shall be provided with prolongations or, at the manufacturer's option, a forging may be selected."

In Specifications A 18 – 21 and A 20 – 21 change Section 17 (*a*), and in Specifications A 19 – 21 and A 63 – 21 change Section 19 (*a*) from their present form: namely,

> "The inspector representing the purchaser shall have free entry at all times while work on the contract of the purchaser is being performed, to all

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

parts of the manufacturer's works which concern the manufacture of the forgings ordered. The manufacturer shall afford the inspector, free of cost, all reasonable facilities to satisfy him that the forgings are being furnished in accordance with these specifications. Tests and inspection at the place of manufacture shall be made prior to shipment."

to read as follows:

"The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the forgings ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the forgings are being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works."

STANDARD SPECIFICATIONS FOR CARBON-STEEL CAR AND TENDER AXLES

(SERIAL DESIGNATION: A 21 – 18)[1]

A revision in the form of separate tentative specifications entitled "Specifications for Carbon-Steel Car and Tender Axles (A 21 – 25 T)"[2] is intended to replace the present standard specifications.

STANDARD SPECIFICATIONS FOR WELDED AND SEAMLESS STEEL PIPE

(SERIAL DESIGNATION: (A 53 – 24)[1]

Section 1.—Change from its present form: namely,

"These specifications cover 'standard,' 'extra strong,' and 'double extra strong' welded and seamless steel pipe. Pipe ordered under these specifications is intended for bending, flanging and other special purposes."

to read as follows:

"*Scope.*—These specifications cover 'standard weight,' 'extra strong,' and 'double extra strong' welded and seamless steel pipe. Pipe ordered under these specifications is intended for coiling, bending, flanging and other special purposes. Butt-welded pipe is not intended for flanging."

STANDARD SPECIFICATIONS FOR LAP-WELDED AND SEAMLESS STEEL AND LAP-WELDED IRON BOILER TUBES

(SERIAL DESIGNATION: A 83 – 24)[1]

Section 16.—Insert a new Paragraph (*e*) to read as follows, relettering the subsequent paragraph accordingly:

"(*e*) The gaging tolerances in Paragraphs (*b*), (*c*) and (*d*) apply only to tubes as rolled or drawn and before swaging and expanding."

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

[2] See p. 509.

STANDARD SPECIFICATIONS FOR WELDED WROUGHT-IRON PIPE

(SERIAL DESIGNATION: A 72 - 24)[1]

Section 1.—Change from its present form: namely,

"These specifications cover 'standard,' 'extra strong' and 'double extra strong' welded wrought-iron pipe."

to read as follows:

"These specifications cover 'standard weight,' 'extra strong' and 'double extra strong' welded wrought-iron pipe. Pipe ordered to these specifications is intended for coiling, bending, flanging, and other special purposes. Butt-welded pipe is not intended for flanging."

Section 3 (b).—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"(*b*) All pipe [3 in. or under] *under 2 in.* in nominal diameter may be butt-welded, unless otherwise specified. All pipe [over 3 in.] *2 in. or over* in nominal diameter shall be lap welded."

Section 9.—Add a new Section 9 to follow the present Section 8 to read as follows, renumbering the subsequent sections accordingly:

"In case of doubt as to the presence of steel, the purchaser may, at his own expense, make micrographic examination and chemical analysis to assist in determining whether the material meets the requirements of Section 3 (*a*)."

STANDARD SPECIFICATIONS FOR STAYBOLT, ENGINE-BOLT AND EXTRA-REFINED WROUGHT-IRON BARS

(SERIAL DESIGNETION: A 84 - 24)[1]

Section 7.—Add two new Paragraphs (*b*) and (*c*) to read as follows, re-lettering the present Paragraph (*b*) to Paragraph (*d*):

"(*b*) *Grade A.*—For bars 1¼ sq. in. in sectional area, the following deductions from the minimum requirements specified in Section 6 (*a*) shall be made for each square inch of nominal section above 1¼ sq. in.:

Tensile strength, 250 lb., but not under 46,000 lb.
Reduction of area, 3 per cent, but not under 40 per cent.

"(*c*) *Grade A.*—For flats of all sizes, the minimum reduction of area shall be 40 per cent."

STANDARD SPECIFICATIONS FOR MALLEABLE CASTINGS

(SERIAL DESIGNATION: A 47 - 24)[2]

Section 3.—Add to the table of requirements a requirement on minimum yield point, as follows:

"Yield point, lb. per sq. in., minimum........ 30 000"

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. C. C. Osterhout, Secretary of Committee A-2 on Wrought Iron, Rome Iron Mills, Inc., Rome, N. Y.

[2] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. Enrique Touceda, Secretary of Committee A-7 on Malleable Castings, 943 Broadway, Albany, N. Y.

Add a new paragraph (*b*) to read as follows:

"(*b*) The yield point shall be determined by the drop of the beam of the testing machine."

RECOMMENDED PRACTICE FOR HEAT TREATMENT OF CASE-HARDENED CARBON-STEEL OBJECTS

(SERIAL DESIGNATION: A 37 – 14)[1]

A revision in the form of a separate tentative recommended practice entitled "Recommended Practice for Carburizing and Heat Treatment of Carburized Objects (A 37 – 25 T)"[2] is intended to replace the present recommended practice.

B. NON-FERROUS METALS

STANDARD SPECIFICATIONS FOR LAKE COPPER WIRE BARS, CAKES, SLABS, BILLETS, INGOTS, AND INGOT BARS

(SERIAL DESIGNATION: B 4 – 13)[3]

Section 4 (*a*).—Change the first sentence in the second paragraph to read as follows by the addition of the italicized figures and the omission of those in brackets:

"Low Resistance Lake wire bars shall have a resistivity not to exceed [0.15535] *0.15436* international ohms per meter-gram at 20° C. (annealed)."

Section 5 (*a*).—Change to read as follows by the addition of the italicized figures and the omission of those in brackets:

"Low Resistance Lake copper shall have a purity of at least [99.880] *99.900* per cent as determined by electrolytic assay, silver being counted as copper."

Section 5 (*b*).—Change the first sentence to read as follows by the addition of the italicized figures and the omission of those in brackets:

"High Resistance Lake copper shall have a purity of at least [99.880] *99.900* per cent, copper, silver, and arsenic being counted together."

Section 7.—Add a new Section 7 to read as follows, renumbering the subsequent sections accordingly:

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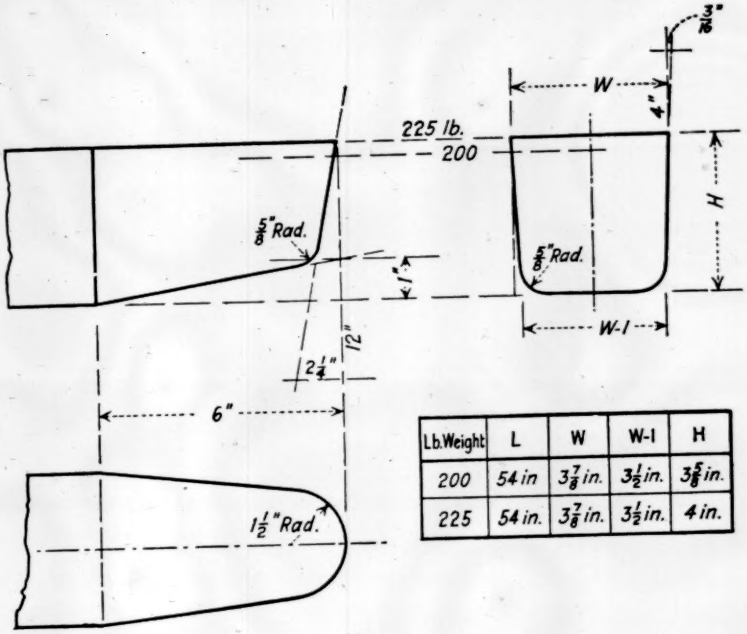
[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. J. H. Hall, Secretary of Committee A-4 on Heat Treatment of Iron and Steel, Taylor-Wharton Iron and Steel Co., High Bridge, N. J.

[2] See p. 568.

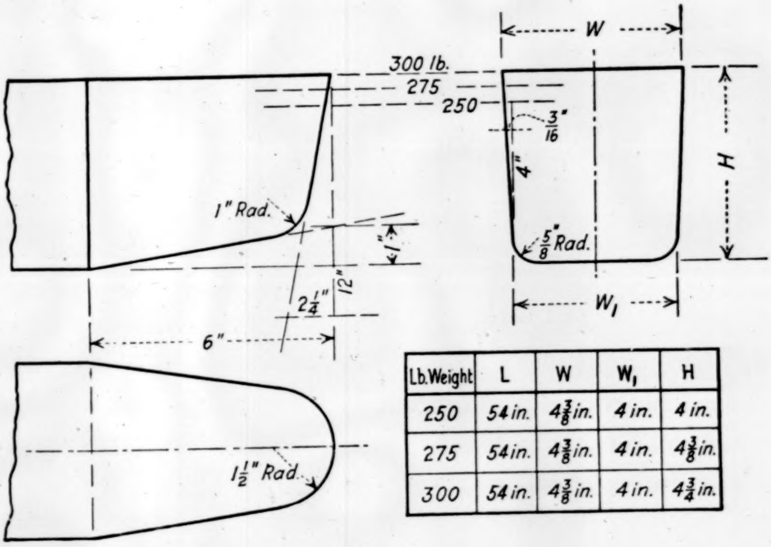
[3] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.



3"/16
W
4"
225 lb.
200
H
5/8" Rad.
5/8" Rad.
1"
W-1
12"
2 1/4"
6"
1 1/2" Rad.

Lb. Weight	L	W	W-1	H
200	54 in	$3\frac{7}{8}$ in.	$3\frac{1}{2}$ in.	$3\frac{5}{8}$ in.
225	54 in.	$3\frac{7}{8}$ in.	$3\frac{1}{2}$ in.	4 in.

FIG. 1.



W
300 lb.
275
250
3"/16
4"
H
1" Rad.
5/8" Rad.
1"
12"
2 1/4"
W1
6"
1 1/2" Rad.

Lb. Weight	L	W	W_1	H
250	54 in.	$4\frac{3}{8}$ in.	4 in.	4 in.
275	54 in.	$4\frac{3}{8}$ in.	4 in.	$4\frac{3}{8}$ in.
300	54 in.	$4\frac{3}{8}$ in.	4 in.	$4\frac{3}{4}$ in.

FIG. 2.

"7. *Standard Sizes and Shapes for 200 to 300-lb. Wire Bars.*—(*a*) One mold shall be used for casting 200 to 230-lb. wire bars, the bottom width of this bar to be 3½ in., the listed weights being 200 and 225 lb. (See Fig. 1.)

"(*b*) One mold shall be used for casting 240 to 300-lb. wire bars, the bottom width of this bar to be 4 in., the listed weights being 250, 275 and 300 lb. (See Fig. 2.)

"(*c*) The length dimension of all bars shall be 54 in. The side draft or taper shall be ⅜ in. in 4 in. (3/16 in. in 4 in. on each side of the bar). The radius of the corners at the bottom of the bars shall be ⅝ in. The end taper at the bottom shall be 6 in. long overall and approximately 2 in. per ft.

"The end taper of the side shall be approximately 2¼ in. per ft. and the end of the bar shall be approximately 3⅜ in. deep at the point."

Section 9.—Change the second paragraph from its present form: namely,

"In a question of metal contents each party shall select a sample of two pieces. These shall be drilled in the presence of both parties, several holes approximately ½ in. in diameter being drilled completely through each piece; scale from set shall be rejected. No lubricant shall be used and drilling shall not be forced sufficiently to cause oxidation of chips. The resulting samples shall be cut up, mixed, and separated into three parts, each of which shall be placed in a sealed package, one for each party and one for the umpire if necessary. Each party shall make an analysis, and if the results do not establish or dismiss the claim to the satisfaction of both parties the third sample shall be submitted to a mutually agreeable umpire, who shall determine the question of fact, and whose determination shall be final."

to read as follows:

"In a question of metal contents, each party shall select a sample of three pieces from the consignment or lot to be investigated. These shall be sampled in the presence of both parties by drilling five holes, approximately ½ in. in diameter, at points equally spaced between the ends of the pieces. With bars, billets, ingots, and ingot bars, these holes should be along a middle line and with slabs and cakes on a diagonal line projected between opposite corners. The drilling shall be from top to bottom and completely through each piece. Scale from set and any surface dirt shall be rejected. No lubricant shall be used and drilling shall not be forced sufficiently to cause oxidation of the chips. In the case of sections having a depth greater than 5 in., drillings may be made from both top and bottom for a depth of not less than 2 in. in each direction instead of completely through, but the drilling shall be otherwise conducted as before described.

"The resulting sample shall be cut up, mixed and separated into three equal portions, each of which shall be placed in a sealed package, one for each party and the third for the umpire, if necessary.

"Each party shall make an analysis by the Standard Method of Battery Assay of Copper (B 34 - 20) of the American Society for Testing Materials and if the results do not establish or dismiss the claim to the satisfaction of both parties concerned, the third sample shall be submitted to a mutually agreeable umpire, who shall determine the question of fact and whose determination shall be final."

STANDARD SPECIFICATIONS FOR ELECTROLYTIC COPPER WIRE BARS, CAKES, SLABS, BILLETS, INGOTS, AND INGOT BARS

(SERIAL DESIGNATION: B 5 – 13)[1]

Sections 3 (*a*).—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> "*Metal Content.*—The copper in all shapes shall have a purity of at least [99.880] *99.900* per cent, as determined by [electrolytic assay] *the A.S.T.M. Standard Method of Battery Assay of Copper* (*B 34 – 20*), silver being counted as copper."

Section 3 (*b*).—Change to read as follows by the addition of the italicized figures and the omission of those in brackets:

> "*Resistivity.*—All wire bars shall have a resistivity not to exceed [0.15535] *0.15436* international ohms per meter-gram at 20° C. (annealed); all ingots and ingot bars shall have a resistivity not to exceed 0.15694 international ohms per meter-gram at 20° C. (annealed)."

Section 6.—Add a new Section 6 to agree with the new Section 7 added to Specifications B 4 – 13 above, renumbering the subsequent sections accordingly.

Section 7.—Change the second paragraph from its present form: namely,

> "In a question of metal contents each party shall select a sample of two pieces. These shall be drilled in the presence of both parties, several holes approximately ½ in. in diameter being drilled completely through each piece; scale from set shall be rejected. No lubricant shall be used and drilling shall not be forced sufficiently to cause oxidation of chips. The resulting samples shall be cut up, mixed, and separated into three parts, each of which shall be placed in a sealed package, one for each party and one for the umpire if necessary. Each party shall make an analysis, and if the results do not establish or dismiss the claim to the satisfaction of both parties the third sample shall be submitted to a mutually agreeable umpire, who shall determine the question of fact, and whose determination shall be final."

to read as follows:

> "In a question of metal contents, each party shall select a sample of three pieces from the consignment or lot to be investigated. These shall be sampled in the presence of both parties by drilling five holes approximately ½ in. in diameter, at points equally spaced between the ends of the pieces. With bars, billets, ingots, and ingot bars, these holes should be along a middle line and with slabs and cakes on a diagonal line projected between opposite corners. The drilling shall be from top to bottom and completely through each piece. Scale from set and any surface dirt shall be rejected. No lubricant shall be used and drilling shall not be forced sufficiently to cause oxidation of the chips. In the case of sections having a depth greater than 5 in., drillings may be made from both top and bottom for a depth of not less than 2 in. in each direction instead

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

of completely through, but the drilling shall be otherwise conducted as before described.

"The resulting sample shall be cut up, mixed and separated into three equal portions, each of which shall be placed in a sealed package, one for each party and the third for the umpire, if necessary.

"Each party shall make an analysis and if the results do not establish or dismiss the claim to the satisfaction of both parties concerned, the third sample shall be submitted to a mutually agreeable umpire, who shall determine the question of fact and whose determination shall be final."

STANDARD SPECIFICATIONS FOR LIGHT ALUMINUM CASTING ALLOYS

(SERIAL DESIGNATION: B 26 – 21)[1]

A revision in the form of separate tentative specifications entitled "Specifications for Aluminum Base Alloy Sand Castings (B 26 – 25 T)"[2] is intended to replace the present standard specifications.

STANDARD SPECIFICATIONS FOR HIGH-STRENGTH BRONZE TROLLEY WIRE, ROUND AND GROOVED: 40 AND 65 PER CENT CONDUCTIVITY

(SERIAL DESIGNATION: B 9 – 16)[3]

A revision in the form of separate tentative specifications entitled "Specifications for Bronze Trolley Wire (B 9 – 25 T)"[4] is intended to replace the present standard specifications.

STANDARD SPECIFICATIONS FOR SEAMLESS ADMIRALTY CONDENSER TUBES AND FERRULE STOCK

(SERIAL DESIGNATION: B 44 – 24)[1]

Section 12.—Insert under Sub-Heading IV on Permissible Variations in Dimensions, a new Section 12 to read as follows, renumbering the subsequent sections accordingly:

"12. ***Diameter.***—The outside diameter of the tubes shall not vary from that specified by more than the following amounts, as measured by 'go' and 'no go' ring gages:

NOMINAL DIAMETER, IN.	PERMISSIBLE VARIATION, IN.
Up to 0.500, inclusive	plus or minus 0.002
Over 0.500 to 0.740, inclusive	" " " 0.0025
Over 0.740 to 1.000, "	" " " 0.003
Over 1.000 to 1.250, "	" " " 0.0035
Over 1.250 to 1.500, "	" " " 0.004

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 574.

[3] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. J. A. Capp, Chairman of Committee B-1 on Copper Wire, General Electric Co., Schenectady, N. Y.

[4] See p. 578.

STANDARD SPECIFICATIONS FOR HIGH SHEET BRASS

(SERIAL DESIGNATION: B 36 – 21)[1]

Table I.—Change Table I in Section 13, covering permissible variations in thickness, to agree with the accompanying Table I.

TABLE I.—PERMISSIBLE VARIATIONS IN THICKNESS OF HIGH SHEET BRASS, IN.

Thickness		Width, in.			
B. & S. Gage No.	Inch	Up to 6 in., inclusive	Over 6 to 9 in., inclusive	Over 9 to 14 in., inclusive	Over 14 to 20 in., inclusive
0000	0.460	0.0043	0.0045	0.0047	0.0049
000	0.4096	0.0042	0.0044	0.0046	0.0048
00	0.3648	0.0041	0.0043	0.0045	0.0047
0	0.3249	0.0040	0.0042	0.0044	0.0046
1	0.2893	0.0039	0.0041	0.0043	0.0045
2	0.2576	0.0038	0.0040	0.0042	0.0044
3	0.2294	0.0037	0.0039	0.0041	0.0043
4	0.2043	0.0036	0.0038	0.0040	0.0042
5	0.1819	0.0035	0.0037	0.0039	0.0041
6	0.1620	0.0034	0.0036	0.0038	0.0040
7	0.1443	0.0033	0.0035	0.0037	0.0039
8	0.1285	0.0032	0.0034	0.0036	0.0038
9	0.1144	0.0031	0.0033	0.0035	0.0037
10	0.1019	0.0030	0.0032	0.0034	0.0036
11	0.0907	0.0029	0.0031	0.0033	0.0035
12	0.0808	0.0028	0.0030	0.0032	0.0034
13	0.0719	0.0027	0.0029	0.0031	0.0033
14	0.0640	0.0026	0.0028	0.0030	0.0032
15	0.0570	0.0025	0.0027	0.0029	0.0031
16	0.0508	0.0024	0.0026	0.0028	0.0030
17	0.0452	0.0023	0.0025	0.0027	0.0029
18	0.0403	0.0022	0.0024	0.0026	0.0028
19	0.0359	0.0021	0.0023	0.0025	0.0026
20	0.0320	0.0020	0.0021	0.0023	0.0024
21	0.0284	0.0019	0.0020	0.0021	0.0022
22	0.0253	0.0018	0.0019	0.0020	0.0021
23	0.0225	0.0017	0.0018	0.0019	0.0020
24	0.0201	0.0016	0.0017	0.0018	0.0019
25	0.0179	0.0015	0.0016	0.0017	0.0018
26	0.0159	0.0014	0.0015	0.0016	0.0017
27	0.0142	0.0013	0.0014	0.0015	0.0016
28	0.0126	0.0012	0.0013	0.0014	0.0015
29	0.0112	0.0011	0.0012	0.0013	0.0014
30	0.0100	0.0011	0.0012	0.0013	0.0014
31	0.0089	0.0010	0.0011	0.0012	0.0013
32	0.0079	0.0010	0.0011	0.0012	0.0013
33	0.0070	0.0009	0.0010	0.0011	0.0012
34	0.0063	0.0009	0.0010	0.0011	0.0012
35	0.0056	0.0008	0.0009	0.0010	0.0011
36	0.0050	0.0008	0.0009	0.0010	0.0011

STANDARD SPECIFICATIONS FOR NAVAL BRASS RODS FOR STRUCTURAL PURPOSES

(SERIAL DESIGNATION: B 21 – 19)[1]

Section 6.—Add a paragraph to read as follows:

"(*b*) Yield point in tension shall be determined as the stress producing an elongation under load of 0.5 per cent, that is, 0.01 in. in a gage length of 2 in."

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

C. CEMENT, LIME, GYPSUM AND CLAY PRODUCTS

STANDARD SPECIFICATIONS AND TESTS FOR PORTLAND CEMENT

(SERIAL DESIGNATION: C 9 - 21)[1]

A revision in the form of a separate tentative specification entitled "Specifications and Tests for Compressive Strength of Portland Cement Mortars (C 9 - 16 T)"[2] is intended to become a part of the present standard specifications.

STANDARD SPECIFICATIONS FOR CEMENT-CONCRETE SEWER PIPE

(SERIAL DESIGNATION: C 14 - 24)[3]

Section 1.—Add the following footnote:

"*Caution.*—The consumer or purchaser is cautioned against using cement-concrete pipe where the sewage shows an acid reaction."

STANDARD SPECIFICATIONS FOR FIRE TESTS OF MATERIALS AND CONSTRUCTION

(SERIAL DESIGNATION: C 19 - 18)[4]

Section 2 (a).—Change to read as follows by the addition of the italicized words and the omission of the words in brackets:

"The temperature fixed by the curve shall be deemed to be the average [true] temperature [of the furnace gases as] obtained from the readings of several thermo-couples (not less than three) symmetrically disposed and distributed [in such a manner as] to show [temperatures of the gases] *the temperature* near all parts of the sample, *said thermo-couples being enclosed in sealed porcelain tubes of ¾-in. outside diameter and ⅛-in. wall thickness, the immersion of the couple in the furnace chamber to be not less than 12 in. Other types of protecting tubes or pyrometers may be used, that under test conditions give the same indications as the above standard within the limit of accuracy that applies for furnace temperature measurements.*

"*For greater difference of design and size the time temperature curve followed shall be modified to give an exposure equal to that obtained by using the standard pyrometer and curve above described.*"

Section 6.—Change to read as follows by the addition of the italicized words and the omission of the words in brackets:

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. B. Young, Secretary of Committee C-1 on Cement, Hydro-Electric Power Commission of Ontario, 8 Strachan Ave., Toronto, Ont., Canada.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part I, p. 599 (1920); also 1925 Book of A.S.T.M. Tentative Standards, p. 193.

[3] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. E. S. Rankin, Secretary of Committee C-4 on Clay and Cement-Concrete Sewer Pipe, Engineer in Charge, Bureau of Sewers, City Hall, Newark, N. J.

[4] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. P. Miller, Secretary of Committee C-5 on Fireproofing, 342 Madison Ave., New York City.

"The fire test on the sample with its applied load, if any, shall be continued until failure occurs, or until it has withstood the test conditions for a period equal to [1¼ times that for which classification is desired] *that herein specified in the Conditions of Acceptance for the given type of construction.*"

Section 7.—Change to read as follows by the addition of the italicized words and the omission of the words in brackets:

"A second test with duplicate sample shall be made to determine the effect of a hose stream on a sample under fire test, the water being applied at the end of a period equal to [three-fourths] *one-half* of that for which classification is desired, but not later than one hour after the beginning of the test; except that for classification periods of one-half hour or less the fire stream test may be omitted."

TABLE II.

Parts of Structure	Type of Protection	Size of Hose Nozzle, in.	Water Pressure at Nozzle, lb.	Time of Application, min.
Floors and Roofs	4 hour	1 1/8	50	[10] *7*
	2 "	1 1/8	50	[5] *4*
	1 "	1 1/8	50	[2.5] *2*
	1/2 "	1 1/8	30	1
	1/4 "	1 1/8	[15] *30*	1
Walls, [Columns] and Partitions	4 "	1 1/8	50	5
	2 "	1 1/8	30	3
	1 "	1 1/8	30	[2.5] *2*
	1/2 "	1 1/8	30	1
	1/4 "	1 1/8	[15] *30*	1
Columns	*4* "	*1 1/8*	*50*	*3*
	2 "	*1 1/8*	*30*	*2*
	1 "	*1 1/8*	*30*	*1 1/2*
	1/2 "	*1 1/8*	*30*	*1*
	1/4 "	*1 1/8*	*30*	*1/2*

Table I.—Change Table I, referred to in Section 8, to read as indicated in the accompanying Table II by the addition of the italicized word and figures and the omission of the word and figures in brackets.

Section 9.—Change to read as follows by the addition of the italicized words and the omission of the words in brackets, changing the side heading from "Loading" to "Loading and Additional Tests":

"For any material or construction intended to carry load other than its own weight, the full [rated safe] working load shall be applied during the entire fire test, also during the fire stream test. After completion of the fire stream test, the sample shall be subjected to [excess loading as prescribed under specifications for the different structural parts] *additional tests as herein specified in the Conditions of Acceptance for the given type of construction.*"

Add a new Section 10, under the sub-division "Conduct of Fire Tests," entitled "Time of Testing," to read as follows:

"The material or construction shall not be tested until a large proportion of its final strength has been attained and the excess of free water has been given off. Where this cannot be attained within 40 days, artificial drying with air temperature not exceeding 100° F. shall be used."

Section 10.—Renumber the present Section 10 to Section 12.

Section 11.—Omit this section reading as follows:

"The floor or roof may be tested as soon after construction as desired, but within 40 days. Artificial drying will be allowed if desired."

Section 12.—Transfer this section, placing it under the subdivision "Conduct of Fire Tests," renumbering it Section 11.

Section 14.—Change the side heading to read "Conditions of Acceptance."

Section 14 (a).—Change to read as follows by the omission of the words in brackets:

"The floor or roof shall have sustained safely the full [rated safe] working load during the fire test without passage of flame for a period equal to 1¼ times that for which classification is desired."

Section 18.—Change the side heading to read "Conditions of Acceptance."

Add five new sections relating to column tests to read as follows:

19. *Size of Sample.*—For column tests, the length of the column exposed to fire shall approximate the clear length of the member in the structure, and for building columns shall be not less than 9 ft. The outside lateral dimensions shall be the minimum for which classification is desired. The details of construction, and exposed connections and protection, if any, shall be applied according to the methods of acceptable field practice.

20. *Loading.*—During the fire test and the fire and water test, a constant working load shall be applied to the column, calculated according to the governing design regulations. Provisions shall be made for transmitting the load to the exposed portion without unduly increasing the effective column length.

21. *Measurements.*—Measurements shall be made of the expansion or depression at the head of the column and determination of the period of expansion. Such other measurements shall be made of deformation and of temperature of the column as will give the most useful information for the given column type.

22. *Position of Nozzle.*—The distance of the nozzle from the column during the application of water shall be 20 ft., the water being applied in succession, as uniformly as possible, over the full height on three of its sides.

23. *Conditions of Acceptance.*—The test shall not be regarded as successful unless the following conditions are met:

(*a*) The column shall have sustained the full working load during the fire test for a period equal to —[1] times that for which classification is desired.

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[1] The quantity to be inserted in this blank space will be supplied by the committee, when the information is available.

(*b*) The column shall have sustained the full working load during the fire stream test as prescribed in Sections 7 and 8, and after its completion and when cold shall sustain a total load equal to the dead load component plus twice the live load component of its design working load.

(*c*) After the fire stream test the column when dry shell withstand, under working load, a fire test of duration equal to —[1] of the period for which classification is desired, but not exceeding one hour.

STANDARD SPECIFICATIONS FOR PAVING BRICK

(SERIAL DESIGNATION: C 7 – 15)[2]

In the Appendix to the specifications, eliminate one of the recognized sizes and types of paving brick, making the recommendations for sizes read as follows:

	WIDTH, IN.	DEPTH, IN.	LENGTH, IN.
Plain wire-cut brick (vertical fiber, lugless).......	3[a]	4[a]	8½
Plain wire-cut brick (vertical fiber, lugless).......	3½[a]	4[a]	8½
Repressed lug brick...........................	3½	4	8½
Wire-cut lug brick (Dunn).....................	3½	4	8½

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[a] These bricks are customarily laid in the pavement to give a depth of 3 or 3½ in., respectively, by 4 in. in width and 8½ in. in length.

STANDARD SPECIFICATIONS FOR HYDRATED LIME FOR STRUCTURAL PURPOSES

(SERIAL DESIGNATION: C 6 – 24)[3]

Section 12.—Change the second and fifth paragraphs from their present form: namely,

> "The lime shall be made into a stiff putty with water and permitted to soak over night. It shall be molded in a rubber ring such as is used with a Vicat needle, resting the specimen on a glass plate."
>
> "If the penetration is less than standard, the sample may be removed from the mold, mixed with more water, and retested. If the penetration is more than standard, the sample shall be discarded and a new one prepared."

to read respectively as follows:

> "The lime shall be made into a stiff putty with water, stirred vigorously with a trowel or spatula for three minutes and permitted to soak over night. It shall be stirred vigorously with a trowel or spatula for three minutes, molded in a rubber ring such as is used with a Vicat needle, resting the specimen on a glass plate."

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[1] The quantity to be inserted in this blank space will be supplied by the committee, when the information is available.

[2] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. W. E. Emley, Secretary of Committee C-3 on Brick, U. S. Bureau of Standards.

[3] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

"If the penetration is less than standard, the sample may be removed from the mold, mixed with more water, stirred vigorously with a trowel or spatula for three minutes, and retested. If the penetration is more than standard, the sample shall be discarded and a new one prepared."

STANDARD METHODS OF TESTING GYPSUM AND GYPSUM PRODUCTS

(Serial Designation: C 26 – 23)[1]

Add the following sections to the present Standard Methods:

XII. Compressive Strength of Gypsum Partition Tile or Block

23. (*a*) Not less than ten full-size gypsum tile specimens shall be used.

(*b*) The test specimens shall be dried to constant weight at a temperature not exceeding 110° F. (43° C.), until two successive weighings of the same specimen do not show a variation in excess of 0.5 per cent. At the option of the manufacturer or purchaser, strength tests may be conducted on samples that are dried at room temperature. However, in cases of controversy all test samples shall be dried to constant weight as herein prescribed.

(*c*) The tile or block shall be tested in the position in which they are designed to be used and shall be bedded on and capped with a felt gasket not less than ⅛ in. nor more than ¼ in. in thickness. At the option of the manufacturer or purchaser, or in cases of controversy, the test samples may be suitably bedded and capped with neat gypsum mortar, or the bearing surfaces of the tile may be planed or rubbed smooth and true. When neat gypsum mortar is used for bedding and capping, the test may be conducted after the mortar has set, but not sooner than one hour after the sample has been capped.

(*d*) The loading head shall completely cover the bearing area of the tile, and the applied load shall be transmitted through a spherical bearing block of proper design. The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute.

XIII. Transverse Strength of Gypsum Partition Tile or Block

24. (*a*) Not less than ten full-size gypsum tile specimens shall be used.

(*b*) The test specimens shall be dried to constant weight as prescribed for the determination of the compressive strength.

(*c*) The tile or block shall be supported on its face (flat) on fixed parallel bearings spaced 24 in. (600 mm.) on centers, and shall be loaded through a similar bearing midway between the supports. All bearing and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of ½ in. (12.7 mm.).

(*d*) The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute. The modulus of rupture shall be calculated in pounds per square inch from the formula:

$$\text{Modulus of Rupture} = \frac{3wl}{2bd^2}$$

where l = distance between supports in inches;
b = breadth (width) of tile in inches;
d = thickness of tile in inches;
w = load in pounds at failure of test specimen.

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. W. E. Emley, Chairman of Committee C-11 on Gypsum, U. S. Bureau of Standards.

XIV. ABSORPTION OF GYPSUM PARTITION TILE OR BLOCK

25. (*a*) The absorption shall be determined upon a test specimen of partition tile or block consisting of approximately one-half of a full-size tile.

(*b*) The test specimen shall be dried to constant weight as prescribed for the determination of the compressive strength.

(*c*) The dried test specimen shall be submerged in water until a constant saturated weight is attained, but in no event for a period of less than two hours, the water temperature being maintained at between 70 and 80° F. (21 and 27° C.). The test specimen shall be removed, the surface wiped off with a damp cloth and the specimen weighed immediately. The percentage of absorption shall be calculated on the dry weight from the formula:

$$\text{Percentage of Absorption} = \frac{100\ (B-A)}{A}$$

where A = weight of dry test specimen;

B = weight of saturated test specimen.

(*d*) The rate of absorption shall be determined by sealing upon the face of the test specimen, which shall first be dried to constant weight as prescribed for determination of the compressive strength, a glass tube 1½ in. in diameter and 12 in. in length, graduated in centimeters and located centrally over a core. Into this tube shall be poured 250 cc. of water at a temperature of between 70 and 80° F. (21 and 27° C.). The rate of absorption shall be reported in cubic centimeters per minute.

XV. TRANSVERSE STRENGTH OF GYPSUM BOARDS

26. (*a*) Test specimens shall be taken from not less than ten gypsum boards.

(*b*) Strength test specimens shall be 12 in. (300 mm.) in width and approximately 18 in. (460 mm.) in length. They shall be supported on fixed parallel bearings spaced 16 in. (410 mm.) on centers, and shall be loaded through a similar bearing midway between the supports. All bearing and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of ⅛ in. (3.2 mm.).

(*c*) Test loads shall be applied at a uniform rate of 60 lb. (27.22 kg.) per minute with a permissible variation of ±10 per cent in the rate. Strength test results shall be reported when the load is applied across the fiber of the surfacing and also parallel to the fiber of the surfacing.

D. MISCELLANEOUS MATERIALS

STANDARD SPECIFICATIONS FOR TURPENTINE

(SERIAL DESIGNATION: D 13 – 24)[1]

Title.—Change to read as follows:

"Specifications for Gum Spirits of Turpentine and for Steam-Distilled Wood Turpentine."

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

"These specifications apply both to the *spirits of* turpentine that is distilled from pine oleo-resins, commonly known as 'gum spirits' or 'spirits of turpentine,' and to turpentine commonly known as '*steam-distilled* wood turpentine' [which is obtained from resinous wood, whether by steam or by destructive distillation]. When ordering under these specifications, the purchaser shall specify whether (a) gum spirits *of turpentine* or (b) *steam-distilled* wood turpentine is desired. [When wood turpentine is specified, it may be stated whether steam or destructively distilled wood turpentine shall be furnished.]"

Section 6.—Under "Refractive index at 20° C." and under "Residue after Polymerization" change "Wood turpentine" to read "Steam-distilled wood turpentine."

STANDARD SPECIFICATIONS FOR BROKEN SLAG FOR WATERBOUND BASE AND WEARING COURSE

(SERIAL DESIGNATION: D 65 – 23)[1]

Section 4.—Omit this section, reading as follows, renumbering the succeeding sections accordingly:

"The percentage of wear shall be not more than 12.0 per cent."

Section 6.—Change the last line of this section to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"[Retained on] *Passing* 2½-in. screen . . . not [less] *more* than [85] *15* per cent."

Section 7 (b).—Omit this paragraph which refers to the method of testing the percentage of wear.

STANDARD SPECIFICATIONS FOR SHOVEL-RUN OR CRUSHER-RUN BROKEN SLAG FOR WATERBOUND BASE

(SERIAL DESIGNATION: D 66 – 23)[2]

Section 4.—Omit this section, reading as follows, renumbering the succeeding sections accordingly:

"The percentage of wear shall be not more than 15.0 per cent."

Section 6 (b).—Omit this paragraph which refers to the method of testing the percentage of wear.

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

[2] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

STANDARD METHOD OF TEST FOR DISTILLATION OF BITUMINOUS MATERIALS SUITABLE FOR ROAD TREATMENT

(SERIAL DESIGNATION: D 20 – 18)[1]

Section 4.—Change the first sentence to read as follows by the addition of the italicized word and the omission of the word in brackets:

> "The apparatus shall be set up as shown in Fig. 2, the thermometer being placed so that the top of the bulb is opposite the *bottom* [middle] of the tubulature."

Make the position of the thermometer in the illustration correspond with this change.

Section 5.—Change the second sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> "After adjusting the thermometer, shield, condenser, etc., the distillation is commenced, the rate being regulated that *50 to 70 drops* (*2.5 to 3.5 cc.*) *pass* [1 cc. passes] over every minute."

STANDARD METHODS OF LABORATORY SAMPLING AND ANALYSIS OF COKE

(SERIAL DESIGNATION: D 37 – 24)[2]

On page 1014 of the 1924 Book of A.S.T.M. Standards under "A Small Riffle Sampler," change "No. 10" to read "No. 20." Also change the paragraphs under "Roll Crusher" and under "Abbe Ball Mill or Hard-Steel Diamond Mortar" to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> "*Hard-Steel* Roll Crusher.—For reducing the product passing a No. 4 sieve to pass a [No. 10] *No. 20* sieve."
>
> "Abbe Ball Mill, *Hard-Steel Roll Crusher*, or Hard-Steel Diamond Mortar.—For reducing the product passing a [No. 10] *No. 20* sieve to pass a No. 60 sieve. The porcelain jars for the ball mill should be approximately 9 in. in diameter and 10 in. high. The flint pebbles should be smooth, hard, and well rounded. *The rolls of the hard-steel roll crusher should revolve at the same speed.*"

On page 1016 under "Reduction of Sample," line 9, change "No. 10 sieve" to read "No. 20 sieve;" also change "400 g." in lines 9 and 10 to read "200 g."

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. W. A. Selvig, Secretary of Committee D-5 on Coal and Coke, 4800 Forbes St., Pittsburgh, Pa.

Change the last two sentences of the first paragraph under "Reduction of Sample" from their present form: namely,

> "Reduce the quantity of sample by quartering or riffling to about 50 g. Pass the entire 50-g. portion through a No. 60 sieve, pulverizing any course particles in a diamond mortar, and mix with remainder of sample, preserving the sample for analysis in a rubber-stoppered glass bottle."

to read as follows:

> "Pass all the material through a No. 60 sieve, pulverizing any coarse particles by impact in a hard-steel diamond mortar, and mix with remainder of sample. Reduce the sample through small riffle sampler to about 50 g. and transfer to a rubber-stoppered glass bottle."

Change the last paragraph under "Reduction of Sample" to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> "In case a ball mill is not available for fine grinding, quarter the 5-lb. sample passing a [No. 10] *No. 20* sieve to 200 g. and pulverize to pass a No. 60 sieve *by means of a hard-steel roll crusher in which the rolls revolve at the same speed, or* by impact in a hard-steel diamond mortar. The use of rubbing surfaces such as a disk pulverizer or a bucking board is never permissible for grinding coke."

STANDARD METHODS OF LABORATORY SAMPLING AND ANALYSIS OF COAL

(SERIAL DESIGNATION: D 22 - 24)[1]

On page 983 of the 1924 Book of A.S.T.M. Standards, under "Method of Sampling, (*A*) When Coal Appears Dry," line 12, change "120 g." to read "200 g."; also change "60 g." in line 6 to read "50 g." and on page 984, line 3, change "60 g." to read "about 50 g."

STANDARD METHODS OF SAMPLING AND ANALYSIS OF CREOSOTE OIL

(SERIAL DESIGNATION: D 38 - 24)[2]

A revision in the form of a separate tentative method entitled "Method of Test for Coke Residue of Creosote Oil (D 168 - 23 T)"[3] is intended to become a part of the present standard methods.

STANDARD METHODS OF TESTING MOLDED INSULATING MATERIALS

(SERIAL DESIGNATION: D 48 - 24)[4]

Section 2.—Change the first sentence to read as follows by the addition of the italicized words:

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. W. A. Selvig, Secretary of Committee D-5 on Coal and Coke, 4800 Forbes St., Pittsburgh, Pa.

[2] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. J. A. Newlin, Secretary of Committee D-7 on Timber, Forest Products Laboratory, Madison, Wis.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 771 (1923); also 1925 Book of A.S.T.M. Tentative Standards, p. 584.

[4] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

"Any standard testing machine may be used ***provided the error in the loading range does not exceed 1 per cent.***"

Section 3.—Change the second sentence to read as follows by the addition of the italicized word and the omission of those in brackets:

"It shall be molded in a hardened [and ground] steel mold ***ground*** to the dimensions given in Fig. 1."

Section 4.—Omit the second and third paragraphs reading as follows:

"Three specimens shall be tested after heating in an oven for one hour at a temperature which is 10° C. (18° F.) below the distortion point of the material, as determined in accordance with Sections 18 to 21. Each specimen shall be taken from the oven and tested immediately while hot.

"Two specimens shall be tested after they have been entirely immersed in water for 48 hours at normal room temperature. The specimens shall be pulled apart at normal room temperature of about 20° C. (68° F.) after the surface water has been removed by wiping with a dry cloth."

Change the first sentence of Paragraph (*b*) from its present form: namely,

"The test specimen shall be pulled apart at such a speed that the beam can be kept well balanced."

to read as follows:

"The speed of the head of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. (1.27 mm.) per minute with the machine running idle."

Fig. 2.—Change from a cube to a cylinder having a height of 1.25 in. (3.18 cm.) and a diameter of 1.125 in. (2.86 cm.). Omit the words "Manufacturer's Name and 'Test Specimen No. 3' Molded on Top in Small Round Body Raised Letters."

Section 6.—Change from its present form: namely,

"Any standard testing machine may be used. The pressure head used for standard compressive strength tests of cement blocks is satisfactory for this purpose. A sheet of soft annealed copper about $\frac{1}{32}$ in. (1 mm.) thick, or heavy blotting paper, shall be placed above and below the specimen to equalize irregularities."

to read as follows:

"Any standard testing machine may be used provided it is accurate to within 1 per cent for the lowest load for which it is used. One end of the specimen shall bear upon an accurately centered spherical bearing block, located whenever practicable at the top and the metal bearing plates shall be directly in contact with the ends of the test specimen. The pressure heads used for standard compression tests of cement (see Tentative Specifications and Tests for Compressive Strength of Portland-Cement Mortars (Serial Designation: C 9 - 16 T) of the American Society for Testing Materials) are satisfactory for this test."

Section 7.—In the first sentence, change the word "cube" to "cylinder." Add a new sentence to read as follows:

"The ends shall be flat and perpendicular to the axis, being ground to this condition if necessary."

Section 8.—Omit the second and third paragraphs, reading as follows:

"Three specimens shall be crushed after heating for one hour at a temperature which is 10° C. (18° F.) below the distortion point of the material as determined in accordance with Sections 18 to 21. Each specimen shall be taken from the oven and tested immediately while hot.

"Two specimens shall be crushed after immersion in water at normal room temperature for 48 hours, with all surface water wiped off with a dry cloth."

Insert new paragraphs to read as follows:

"Additional tests shall be made at elevated temperatures, the actual temperatures selected depending upon the use that is to be made of the material. Five specimens shall be tested at each temperature selected. The specimen shall be kept at that temperature a sufficient length of time to become of uniform temperature throughout and shall be maintained at that temperature during the test.

"NOTE 1.—A convenient method of making this test is to submerge the specimens in a suitably lagged, electrically heated oil bath of about two gallons capacity. The specimens should be placed in the bath at least one-half hour before testing and the temperature maintained approximately constant at the desired testing temperature until the completion of that test. A pale mineral oil having a viscosity of 100 seconds ± 10 seconds at 38° C. (100° F.) Saybolt is satisfactory for this purpose. The specimens should not be placed in the oil an excessive time in advance of testing. The oil bath should be well stirred to insure uniform temperature throughout. The temperature should be determined by means of a thermocouple sealed in a similar specimen in the bath; or by the use of an A.S.T.M. partial immersion thermometer conforming to the Standard Specifications for Partial-Immersion Thermometer for General Use, −20 to +150° C., 0 to +300° F. (Serial Designation: D 182) of the American Society for Testing Materials,[1] so inserted in a hole in a specimen that the bulb is completely enclosed and sealed in.

"If tests are required over the whole range of temperature rather than at particular points, the method of testing may be found simpler and quicker if modified in accordance with Note 2, below.

"NOTE 2.—The oil with 5 specimens immersed in it should be heated to the highest temperature to be used and allowed to cool slowly. Specimens should be tested in succession and the average temperature during the actual time of testing of each specimen taken as that corresponding to each strength determination. Specimens should be added to the bath successively on such a schedule that each will remain in the bath at least ½ hour before testing. An average curve shall be drawn through the plotted data."

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[1] **A.S.T.M. Standards Adopted in 1925.**

Change the first sentence of Paragraph (*b*) from its present form: namely,

> "The load shall be applied in a direction at right angles to that in which the pressure was applied in molding, and at such a rate of speed that will permit the beam to be kept well balanced from zero load until the specimen is crushed."

to read as follows:

> "The load shall be applied on the ends of the specimen and the cross-head speed of the testing machine shall be such that the beam of the machine can be kept balanced but shall not exceed 0.050 in. (1.27 mm.) per minute when the machine is running idle."

In Paragraph (*e*), change the word "jaws" to read "head." Add a new sentence to read as follows:

> "The time required for testing each specimen should be recorded."

Section 10.—Change to read as follows:

> "Any standard testing machine may be used provided it is accurate to within 1 per cent for the lowest load for which it is used. The distance between points of support shall be 4 in. (10.16 cm.). The supports shall have the contact edges rounded to a radius of ⅛ in. (3.18 mm.). The load shall be applied midway between the supports by a pressure piece, the bearing edge of which shall be rounded to a radius of ⅛ in. (3.18 mm.)."

Section 12.—In the first sentence of Paragraph (*a*) change the words "four specimens" to read "five specimens."

Change the second paragraph from its present form: namely,

> "Three specimens shall be tested after immersion in water at normal room temperature for 48 hours with all surface water wiped off with a dry cloth."

to read as follows:

> "Additional tests shall be made at elevated temperatures as outlined in Section 8."

Change the first two sentences of Paragraph (*b*) from their present form: namely,

> "The load shall be applied at as slow a speed as possible, so that the beam may be kept well balanced from zero load until the first sign of failure. All tests shall be made at room temperature of about 20° C. (68° F.)."

to read as follows:

> "The cross-head speed of the testing machine shall be such that the beam of the machine can be kept balanced but shall not exceed 0.050 in. (1.27 mm.) per minute when the machine is running idle."

Section 13.—Change Paragraph (*e*) to read as follows by the addition of the italicized word and the omission of those in brackets:

"The [amount of] ***maximum*** deflection at the center in inches or in millimeters."

Omit Paragraph (*f*) reading as follows:

"The distance between the supports in inches or in millimeters."

Section 16 (*b*).—Change the second paragraph from its present form: namely,

"Three specimens shall be punctured after heating one hour in an oven heated to 10° C. (18° F.) below the distortion point of the material as determined in Sections 18 to 21. The specimen shall be taken from the oven and immediately punctured in air while hot. This test is intended to cover all molded materials which do not withstand working temperatures above 125° C. (257° F.). Tests on materials which resist high temperatures may be made above 125° C. (257° F.) when required."

to read as follows:

"Additional tests shall be made at elevated temperatures, the actual temperatures selected depending upon the use that is to be made of the material. Five specimens shall be tested at each temperature while in an oven maintained at the proper temperature, either by hand adjustment or by automatic thermostat. Specimens shall be placed in the oven at least ½ hour previous to testing and the temperature shall then be maintained approximately constant at the testing temperature until the completion of that test. Temperature shall be determined by means of a thermocouple or a mercury thermometer placed in the oven close to the specimens and care shall be taken to avoid placing the specimens too close to the heater.

"Specimens with which arcing over the edge is encountered shall be tested under oil as described under Section 16, and in such cases the oil bath containing the specimens shall be installed in the oven and the temperature determined by immersing the thermometer in the oil."

In the third paragraph, change the words "Two specimens" to "Three specimens."

Section 17.—Add a new paragraph to read as follows:

"(*e*) Temperature at which each test was made."

Section 23.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"The test specimen [No. 1] *No. 3* shown in [Fig. 4] *Fig. 2* shall be used for this test. [After the puncture tests prescribed in Section 16 (*b*) are completed on the five specimens, three of them shall be taken and the entire rim of each specimen shall be sawed off with a hack saw ¼ in. (6 mm.) back from the top edge of the specimen so as to expose a more or less uniformly cut surface.]"

Section 24.—Change the first sentence from its present form: namely,

"Weigh each of the three test specimens after the rim has been sawed off."

to read as follows:

"Three specimens shall be tested. Weigh each of the three separately in the as-received condition."

In the fifth and sixth sentences change the words "100 hours" to "48 hours."

Section 25.—In paragraphs (*c*) and (*d*) change the words "100 hours" to " 48 hours."

STANDARD DEFINITIONS OF TERMS RELATING TO PAINT SPECIFICATIONS

(SERIAL DESIGNATION: D 16 - 24)[1]

Add the following three new definitions of terms:

Toughness.—Relative degree of resistance to impact without fracture; apposed to brittleness.

Elasticity of Paint and Varnish Films.—That property which allows a paint or varnish film to follow, without rupture, changes in the extent and form of the surface to which it is applied.

"*Screen* (*Sieve*).—A plate or sheet or woven cloth with regularly spaced apertures of uniform size, mounted on a suitable frame or holder, for use in separating materials according to size."

"NOTE.—The shape and spacing of apertures, size of wires or threads, thickness of plate or sheet, allowable variations and similar properties should be described in the specifications."

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

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VOLUME 25, PART I.

SUBJECT INDEX

A

Absorption.
  Tentative Method of Test for Water Absorption of Slate, 754.

Admiralty Metal.
  Tentative Revision of Standard Specifications for Seamless Admiralty Condenser Tubes and Ferrule Stock, 902.

Aggregates.
  Concrete Aggregate Material Investigation Report. Report of Committee C-9, Appendix II, 209.
  Concrete and Concrete Aggregates. Report of Committee C-9, 199.
  Tentative Definition of the Term Sand, 635.

Alloys.
  Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 150.
  Ferro-Alloys. Report of Committee A-9, 121. Discussion, 123.
  Non-Ferrous Metals and Alloys. Report of Committee B-2, 129. Addendum, 149.
  Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys, 590.
  Tentative Methods of Chemical Analysis of Ferro-Alloys, 542.
  Tentative Methods of Sampling Ferro-Alloys, 537.
  Tentative Revision of Standard Specifications for Light Aluminum Casting Alloys, 902.
  Tentative Specifications for Aluminum Base Alloy Sand Castings, 574.
  Tentative Specifications for Bronze Trolley Wire, 578.
  Tentative Specifications for Ferro-Chromium, 532.
  Tentative Specifications for Ferro-Manganese, 530.
  Tentative Specifications for Ferro-Silicon, 531.
  Tentative Specifications for Ferro-Vanadium, 533.
  Tentative Specifications for Muntz Metal Condenser Tube Plates, 583.
  Tentative Specifications for Non-Ferrous Insect Screen Cloth, 587.
  Tentative Specifications for Spiegeleisen, 529.

Alloy-Steel.
  Tentative Revision of Standard Specifications for Carbon-Steel and Alloy-Steel Forgings, 895.
  Tentative Revision of Standard Specifications for Quenched-and-Tempered Alloy-Steel Axles, Shafts and Other Forgings for Locomotives and Cars, 895.
  Tentative Specifications for Alloy-Steel Bolting Material for High-Temperature Service, 523.
  Tentative Specifications for Structural Silicon Steel, 514.

Aluminum.
  Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys, 590.
  Tentative Revision of Standard Specifications for Light Aluminum Casting Alloys, 902.
  Tentative Specifications for Aluminum Ingots for Remelting, 585.
  Tentative Specifications for Aluminum Base Alloy Sand Castings, 574.
Annual Address.
  Promotion of Knowledge of Engineering Materials. Annual Address by the President, F. M. Farmer, 34.
Annual Meeting.
  Summary of the Proceedings of the Twenty-eighth Annual Meeting, 11.
Anti-Fouling Paints.
  Anti-Fouling Paints. Report of Sub-Committee XXIII, Committee D-1, 244.
Antimony.
  Review of the Antimony Situation by H. K. Masters. Report of Sub-Committee B-2, Appendix, 143.
Asphalt.
  Tentative Methods of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles, 792.
  Tentative Specifications for Acid-Resisting Asphalt Mastic, 759.
  Tentative Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-up Roofs, 770.
  Tentative Specifications for Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles, 765.
  Tentative Specifications for Smooth-Surfaced Asphalt Roll-Roofing, 761.
Axles.
  Tentative Revision of Standard Specifications for Carbon-Steel Car and Tender Axles, 896.
  Tentative Revision of Standard Specifications for Quenched-and-Tempered Alloy-Steel Axles, Shafts and Other Forgings for Locomotives and Cars, 895.
  Tentative Revision of Standard Specifications for Quenched-and-Tempered Carbon-Steel Axles, Shafts and Other Forgings for Locomotives and Cars, 895.
  Tentative Specifications for Carbon-Steel Car and Tender Axles, 509.

B

Bars.
  Tentative Revision of Standard Specifications for Staybolt, Engine-Bolt and Extra-Refined Wrought Iron Bars, 897.
Bituminous Materials.
  Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 342.
  Penetration. Report of Sectional Committee, Committee D-4, 301.
  Results of tests by various investigators on penetration of bituminous materials, 302.
  Tentative Methods of Float Test for Bituminous Materials, 748.
  Tentative Methods of Testing Bituminous Mastics, Grouts, and Like Mixtures, 786.
  Tentative Methods of Testing Cable Splicing and Pothead Compounds, 810.
  Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing, 776.

Bituminous Materials (*Continued*):
  Tentative Methods of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles, 792.
  Tentative Revision of Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment, 911.
  Tentative Specifications for Acid-Resisting Asphalt Mastic, 759.
  Tentative Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs, 770.
  Tentative Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs, 773.
  Tentative Specifications for High-Carbon Tar Cement, 740.
  Tentative Specifications for High-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product), 738.
  Tentative Specifications for Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaces Asphalt Shingles, 765.
  Tentative Specifications for Smooth-Surfaced Asphalt Roll-Roofing, 761.
Boiler Tubes.
  Tentative Revision of Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes, 896.
Bolts.
  Tentative Specifications for Alloy-Steel Bolting Material for High-Temperature Service, 523.
Bone Black.
  Tentative Specifications for Bone Black, 652.
Brass.
  Tentative Revision of Standard Specifications for Naval Brass Rods for Structural Purposes, 903.
  Tentative Revision of Standard Specifications for High Sheet Brass, 903.
Brick.
  Brick. Report of Committee C-3, 184.
  Tentative Revision of Standard Specifications for Paving Brick, 907.
Brinell Hardness.
  Tentative Methods of Brinell Hardness Testing of Metallic Materials, 872.
Bronze.
  Tentative Revision of Standard Specifications for High-Strength Bronze Trolley Wire, Round and Grooved, 40 and 65-per-cent Conductivity, 902.
  Tentative Specifications for Bronze Trolley Wire, 578.
Building Tile.
  See **Tile.**
Burning Quality.
  Tentative Method of Test for Burning Quality of Long-Time Burning Oil for Railway Use, 718.

C

Cable Splicing.
  Tentative Methods of Testing Cable Splicing and Pothead Compounds, 810.
Carburizing.
  Tentative Recommended Practice for Carburizing and Heat Treatment of Carburized Objects, 568.
  Tentative Revision of Standard Recommended Practice for Heat Treatment of Case-Hardened Carbon-Steel Objects, 898.

Case Hardening.
  See **Carburizing.**

Castings.
  Classification of refractories for malleable-cast-iron industry, 195.
  Malleable Castings. Report of Committee A-7, 118. Discussion, 119.
  Tentative Revision of Standard Specifications for Light Aluminum Casting Alloys, 902.
  Tentative Revision of Standard Specifications for Malleable Castings, 897.
  Tentative Specifications for Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service, 519.
  Tentative Specifications for Aluminum Base Alloy Sand Castings, 574.

Cast Iron.
  Cast Iron. Report of Committee A-3, 84.
  Discussion on cast iron test bars, 87.

Cement.
  Brief Bibliography on Portland Cement. Report of Committee C-1, Appendix, 172.
  Cement. Report of Committee C-1, 159.
  Discussion on cement testing, 178.
  Manual of Cement Testing. Report of Committee C-1, Appendix, 161.
  Tentative Method of Test for Consistency of Portland-Cement Concrete, 746.
  Tentative Revision of Standard Specifications and Tests for Portland Cement, 904.
  Training of cement testers, 170.

China Wood Oil.
  See **Tung Oil.**

Chrome Green.
  Tentative Specifications for Pure Chrome Green, 656.
  Tentative Specifications for Reduced Chrome Green, 658.

Chrome Yellow.
  Tentative Specifications for Chrome Yellow, 654.

Cloud Point.
  Proposed Specifications for A.S.T.M. Low-Cloud and Pour Test Thermometers, −60 to +20° C., −70 to +70° F. Report of Committee D-15, 393.
  Tentative Method of Test for Cloud and Pour Points of Petroleum Products, 725.

Coal.
  Coal and Coke. Report of Committee D-5, 307.
  Tentative Definitions of Terms Relating to Coal, 752.
  Tentative Revision of Standard Methods of Laboratory Sampling and Analysis of Coal, 912.

Coal-Tar.
  See **Tar.**

Coke.
  Classification of refractories for by-product coke ovens, 196.
  Coal and Coke. Report of Committee D-5, 307.
  Tentative Revision of Standard Methods of Laboratory Sampling and Analysis of Coke, 911.

Color.
  Tentative Method of Test for Color of Petrolatum by Means of the Union Colorimeter, 713.

Compression Testing.
- Compressive strength of concrete as determined on specimens of various size and shape, 200.
- Tentative Methods of Compression Testing of Metallic Materials, 867.

Concrete.
- Compressive strength of concrete as determined on specimens of various size and shape, 200.
- Concrete Aggregate Material Investigation Report. Report of Committee C-9, Appendix II, 209.
- Concrete and Concrete Aggregates. Report of Committee C-9, 199.
- Digest of Literature on Methods of Making Flexure and Tension Tests of Concrete by Duff A. Abrams. Report of Committee C-9, Appendix I, 205.
- Tentative Method of Securing Specimens of Hardened Concrete from the Structure, 631.
- Tentative Method of Test for Consistency of Portland-Cement Concrete, 746.
- Tentative Revision of Standard Specifications for Cement-Concrete Sewer Pipe, 904.

Condenser Tubes.
- Tentative Revision of Standard Specifications for Seamless Admiralty Condenser Tubes and Ferrule Stock, 902.
- Tentative Specifications for Muntz Metal Condenser Tube Plates, 583.

Consistency.
- Tentative Method of Test for Consistency of Portland-Cement Concrete, 746.
- Tentative Method of Test for Penetration of Greases, 701.

Copper.
- Copper Wire. Report of Committee B-1, 126.
- Tentative Revision of Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars, 901.
- Tentative Revision of Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars, 898.
- Tentative Specifications for Non-Ferrous Insect Screen Cloth, 587.

Corrosion.
- Anti-Fouling Paints. Report of Sub-Committee XXIII, Committee D-1, 244.
- Corrosion of Iron and Steel. Report of Committee A-5, 95.
- Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 150.
- Long-time exposure tests of non-ferrous screen wire materials, 384.
- Outline of atmospheric exposure tests on zinc-coated products, 104.
- Total Immersion Tests. Report of Sub-Committee V, Committee A-5, 99.

Cotton.
- Tentative Specifications for Tolerances and Test Methods for Cotton Sewing Threads, 837.
- Tentative Specifications for Tolerances and Test Methods for Cotton Yarns, Single and Plied, 831.
- Tentative Specifications for Tolerances for Numbered Cotton Duck, 840.

Creosote Oil.
- Tentative Revision of Standard Methods of Sampling and Analysis of Creosote Oil, 912.
- Timber Preservatives. Report of Sub-Committee VI, Committee D-7, 340.

D

Damp-Proofing.
See **Waterproofing.**

Definitions of Terms.
Definitions of Terms Used in Paint Specifications. Report of Sub-Committee VI, Committee D1, 232.
Nomenclature and Definitions. Report of Committee E-8, 488.
Nomenclature. Report of Sub-Committee XX, Committee D2, 287.
Tentative Definition of the Term Sand, 635.
Tentative Definition of the Term Screen (Sieve), 889.
Tentative Definitions of Terms Relating to Coal, 752.
Tentative Definitions of Terms Relating to the Gypsum Industry, 633.
Tentative Definitions of Terms Relating to Methods of Testing, 879.
Tentative Definitions of Terms Relating to Specific Gravity, 887.
Tentative Revision of Standard Definitions of Terms Relating to Paint Specifications, 917.

Dielectric Strength.
Tentative Method of Testing Electrical Insulating Materials for Voltage Effects at Radio Frequencies, 804.
Tentative Methods of Testing Insulating Varnishes, 797.
Tentative Methods of Testing Cable Splicing and Pothead Compounds, 810.

Distillation.
Application of distillation methods for moisture, 421.
Results of tests to determine naptha content of crude oils, 291.
Tentative Method of Test for Distillation of Gasoline, Naptha, Kerosine, and Similar Petroleum Products, 685.
Tentative Method of Test for Distillation of Natural Gas Gasoline, 693.
Tentative Methods of Testing Gas Oils (Gravity, Distillation, Sulfur, Carbon Residue, Pour Point, Viscosity, Water), 732.
Tentative Revision of Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment, 911.
Timber Preservatives. Report of Sub-Committee VI, Committee D-7, 340.

Douglas Fir.
See **Timber.**

Duck.
Tentative Specifications for Tolerances for Numbered Cotton Duck, 840.

E

Emulsification.
Emulsification. Report of Sub-Committee XI, Committee D-2, 272.
Results of investigation on modified A.S.T.M. steam emulsion test, 273.

Executive Committee.
Annual Report of the Executive Committee, 46.

F

Fabrics.
Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing, 776.

Felt.
Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing, 776.
Tentative Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs, 770.
Tentative Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs, 773.

Ferro-Alloys.
Ferro-Alloys. Report of Committee A-9, 121. Discussion, 123.
Tentative Methods of Chemical Analysis of Ferro-Alloys, 542.
Tentative Methods of Sampling Ferro-Alloys, 537.
Tentative Specifications for Ferro-Chromium, 532.
Tentative Specifications for Ferro-Manganese, 530.
Tentative Specifications for Ferro Silicon, 531.
Tentative Specifications for Ferro-Vanadium, 533.
Tentative Specifications for Spiegeleisen, 529.
Tentative Specifications for Tungsten Powder, 528.

Ferro-Chromium.
Tentative Specifications for Ferro-Chromium, 532.

Ferro-Manganese.
Tentative Specifications for Ferro-Manganese, 530.

Ferro-Silicon.
Tentative Specifications for Ferro-Silicon, 531.

Ferro-Vanadium.
Tentative Specifications for Ferro-Vanadium, 533.

Fireproofing.
Tentative Revision of Standard Specifications for Fire Tests of Materials and Construction, 904.

Flexure Testing.
Digest of Literature on Methods of Making Flexure and Tension Tests of Concrete by Duff A. Abrams. Report of Committee C-9, Appendix I, 205.
Proposed Methods of Flexure Testing of Metallic Materials. Report of Committee E-1, Appendix I, 424.
Tentative Methods of Flexure Testing of Slate (Determination of Modulus of Rupture and Modulus of Elasticity), 756.

Float Test.
Tentative Method of Float Test for Bituminous Materials, 748.

Forgings.
Tentative Revision of Standard Specifications for Carbon-Steel and Alloy-Steel Forgings, 895.
Tentative Revision of Standard Specifications for Carbon-Steel Car and Tender Axles, 896.
Tentative Revision of Standard Specifications for Carbon-Steel Forgings for Locomotives, 895.
Tentative Revision of Standard Specifications for Quenched-and-Tempered Alloy-Steel Axles, Shafts and Other Forgings for Locomotives and Cars, 895.
Tentative Revision of Standard Specifications for Quenched-and-Tempered Carbon-Steel Axles, Shafts and Other Forgings for Locomotives and Cars, 895.
Tentative Specifications for Carbon-Steel Car and Tender Axles, 509.

Free Acid.
Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants, 722.

Free Alkali.
Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants, 722.

G

Galvanizing.
Outline of atmospheric exposure tests on zinc-coated products, 104.
Proposed Requirements for Zinc-Coated or Galvanized Wire. Report of Sub-Committee VI, Committee A-5, Appendix, 102.

Gas Oils.
Tentative Methods of Testing Gas Oils (Gravity, Distillation, Sulfur, Carbon Residue, Pour Point, Viscosity, Water), 732.

Gasoline.
Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products, 685.
Tentative Method of Test for Distillation of Natural Gas Gasoline, 693.

Grease.
Grease. Report of Sub-Committee IV, Committee D-2, 266.
Tentative Method of Test for Penetration of Greases, 701.

Grout.
Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures, 786.

Gypsum.
Gypsum. Report of Committee C-11, 211. Discussion, 218.
Proposed Revisions of the Standard Methods of Testing Gypsum and Gypsum Products. Report of Committee C-11, Appendix, 216.
Tentative Definitions of Terms Relating to the Gypsum Industry, 633.
Tentative Revision of Standard Methods of Testing Gypsum and Gypsum Products, 908.

H

Hardness.
Micro-Hardness. Report of Sub-Committee V, Committee E-4, 442.
Tentative Methods of Brinell Hardness Testing of Metallic Materials, 872.

Heat Treatment.
Heat Treatment of Iron and Steel. Report of Committee A-4, 94.
Results of vibratory and tension tests on staybolt iron, annealed and unannealed, 82.
Tentative Recommended Practice for Carburizing and Heat Treatment of Carburized Objects, 568.
Tentative Revision of Recommended Practice for Heat Treatment of Case-Hardened Carbon-Steel Objects, 898.

High Temperature.
Tentative Specifications for Alloy-Steel Bolting Material for High-Temperature Service, 523.
Tentative Specifications for Carbon-Steel Castings for Valves, Flanges and Fittings for High-Temperature Service, 519.

Hydrated Lime.
See **Lime.**

I

Illuminating Oils.

Tentative Method of Test for Burning Quality of Long-Time Burning Oil for Railway Use, 718.

Ingots.

Tentative Specifications for Aluminum Ingots for Remelting, 585.

Insulating Materials.

Electrical Insulating Materials. Report of Committee D-9, 348. Discussion, 369.
Life Test for Transformer Oils by E. A. Snyder and D. C. Cox. Report of Committee D-9, Appendix, 363.
Tentative Method of Testing Electrical Insulating Materials for Voltage Effects at Radio Frequencies, 804.
Tentative Methods of Testing Cable Splicing and Pothead Compounds, 810.
Tentative Methods of Testing Insulating Varnishes, 797.
Tentative Methods of Testing Laminated Sheet Insulating Materials, 825.
Tentative Methods of Testing Untreated Insulating Paper, 815.
Tentative Revision of Standard Methods of Testing Molded Insulating Materials, 912.

Iron.

Cast Iron. Report of Committee A-3, 84.
Corrosion of Iron and Steel. Report of Committee A-5, 95.
Discussion on cast iron test bars, 87.
Heat Treatment of Iron and Steel. Report of Committee A-4, 94.
Inspection of the Fort Sheridan, Pittsburgh, and Annapolis Tests. Report of Sub-Committee III, Committee A-5, 97.
Outline of atmospheric exposure tests on zinc-coated products, 104.
Preparation of Iron and Steel Surfaces for Painting. Report of Sub-Committee XIV, Committee D-1, 238.
Proposed methods of sampling and chemical analysis of wrought iron, 79.
Results of vibratory and tension tests on staybolt iron, annealed and unannealed, 82.
Tentative Revision of Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes, 896.
Tentative Revision of Standard Specifications for Staybolt, Engine-Bolt and Extra-Refined Wrought-Iron Bars, 897.
Tentative Revision of Standard Specifications for Welded Wrought Iron Pipe, 897.
Tentative Specifications for Hollow Staybolt Iron, 534.
Total Immersion Tests. Report of Sub-Committee V, Committee A-5, 99.
Wrought Iron. Report of Committee A-2, 77.

Iron Hydroxide.

Tentative Specifications for Iron Oxide and Iron-Hydroxide, 646.

Iron Oxide.

Tentative Specifications for Iron Oxide and Iron Hydroxide, 646.

K

Kerosine.
  Tentative Method of Test for Burning Quality of Long-Time Burning Oil for Railway Use, 718.
  Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products, 685.

Knit Goods.
  Tentative Specifications for Tolerances and Test Methods for Knit Goods, 842.

L

Lampblack.
  Tentative Specifications for Lampblack, 650.

Lime.
  Lime. Report of Committee C-7, 186.
  Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime, 618.
  Tentative Revision of Standard Specifications for Hydrated Lime for Structural Purposes, 907.
  Tentative Specifications for Hydrated Lime for Use in Water Treatment, 616.
  Tentative Specifications for Quicklime for Use in the Manufacture of Sulfite Pulp, 612.
  Tentative Specifications for Quicklime for Use in Water Treatment, 614.

Linseed Oil.
  Tentative Methods of Routine Analysis of White Linseed Oil Paints, 664.

Lithopone.
  Tentative Specifications for Lithopone, 648.

Lubricants.
  Grease. Report of Sub-Committee IV, Committee D-2, 266.
  Petroleum Products and Lubricants. Report of Committee D-2, 248.
  Tentative Method of Test for Color of Petrolatum by Means of the Union Colorimeter, 713.
  Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants, 722.
  Tentative Method of Test for Penetration of Greases, 701.
  Tentative Methods of Test for Viscosity of Petroleum Products and Lubricants, 707.

M

Malleable Castings.
  Classification of refractories for malleable-cast-iron industry, 195.
  Malleable Castings. Report of Committee A-7, 118. Discussion, 119.
  Tentative Revision of Standard Specifications for Malleable Castings, 897.

Mastic.
  Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures, 786.
  Tentative Specifications for Acid-Resisting Asphalt Mastic, 759.

Mechanical Analysis.
  List of points to be considered in drawing up specifications for a sieving test, Tentative Specifications for Sieves for Testing Purposes, 853.
  Tentative Method of Mechanical Analysis of Subgrade Soils, 742.
  Tentative Specifications for Sieves for Testing Purposes, 850.

Metallography.
  Discussion on X-ray metallography, 470.
  Metallography. Report of Committee E-4, 440. Discussion, 470.
  Micro-Hardness. Report of Sub-Committee V, Committee E-4, 442.
  Tentative Recommended Practice for Thermal Analysis of Steel, 890.
  X-ray Metallography. Report of Sub-Committee VI, Committee E-4, 444.
Mineral Spirits.
  Report on collaborative tests on detection of sulfur in mineral spirits, 222.
Modulus of Elasticity.
  Tentative Methods of Flexure Testing of Slate (Determination of Modulus of Rupture and Modulus of Elasticity), 756.
Modulus of Rupture.
  Tentative Methods of Flexure Testing of Slate (Determination of Modulus of Rupture and Modulus of Elasticity), 756.
Moisture Equivalent.
  Tentative Method of Test for the Determination of Moisture Equivalent of Subgrade Soils in the Field, 745.
Muntz Metal.
  Tentative Specifications for Muntz Metal Condenser Tube Plates, 583.

N

Naphtha.
  Results of tests to determine naphtha content of crude oils, 291.
  Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products, 685.
Natural Gas Gasoline.
  Tentative Method of Test for Distillation of Natural Gas Gasoline, 693.
Neutralization Number.
  Neutralization Number and Saponification. Report of Sub-Committee XIII, Committee D-2, 280.
  Proposed Method of Test for Neutralization Number of Petroleum Oil, Titration by Electrometric Method. Report of Sub-Committee XIII, Committee D-2, Appendix, 282.
  Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants, 722.
Nomenclature.
  See **Definitions of Terms.**

O

Oil.
  See also **Petroleum Products.**
  Calorific Value of Fuel Oil. Report of Sub-Committee XXIV, Committee D-2, 292.
  Life Test for Transformer Oils by E. A. Snyder and D. C. Cox. Report of Committee D-9, Appendix, 363.
  Proposed Method for Determination of Oil in Wax. Report of Sub-Committee III, Committee D-2, Appendix, 263.
  Proposed Method of Test for Neutralization Number of Petroleum Oil, Titration by Electrometric Method. Report of Sub-Committee XIII, Committee D-2, Appendix, 282.

Oil (*Continued*):
  Results of tests to determine naphtha content of crude oils, 291.
  Tentative Method of Test for Burning Quality of Long-Time Burning Oil for Railway Use, 718.
  Tentative Method of Test for Cloud and Pour Points of Petroleum Products, 725.
  Tentative Methods of Testing Gas Oils (Gravity, Distillation, Sulfur, Carbon Residue, Pour Point, Viscosity, Water), 732.
  Tentative Specifications for Raw Tung Oil, 636.
  Timber Preservatives. Report of Sub-Committee VI, Committee D-7, 340.

P

Paint.
  Anti-Fouling Paints. Report of Sub-Committee XXIII, Committee D-1, 244.
  Definitions of Terms Used in Paint Specifications. Report of Sub-Committee VI, Committee D-1, 232.
  Method of Application of Paint by Spraying. Report of Sub-Committee XXII, Committee D-1, 243.
  Physical Properties of Paint Materials. Report of Sub-Committee XVIII, Committee D-1, 241.
  Preparation of Iron and Steel Surfaces for Painting. Report of Sub-Committee XIV, Committee D-1, 238.
  Tentative Method of Routine Analysis of White Linseed Oil Paints, 664.
  Tentative Revision of Standard Definitions of Terms Relating to Paint Specifications, 917.
Paint Vehicles.
  Tentative Specifications for Raw Tung Oil, 636.
  Testing of Paint Vehicles. Report of Sub-Committee III, Committee D-1, 227.
Paper.
  Tentative Methods of Testing Untreated Insulating Paper, 815.
  Tentative Specifications for Quicklime for Use in the Manufacture of Sulfite Pulp, 612.
Paraffin Wax.
  Paraffin Wax. Report of Sub-Committee III, Committee D-2, 260.
Paving Brick.
  Tentative Revision of Standard Specifications for Paving Brick, 907.
Penetration.
  Penetration. Report of Sectional Committee, Committee D-4, 301.
  Results of tests by various investigators on penetration of bituminous materials, 302.
  Tentative Method of Test for Penetration of Greases, 701.
Petrolatum.
  Tentative Method of Test for Color of Petrolatum by Means of the Union Colorimeter, 713.
Petroleum Products.
  Crude Petroleum. Report of Sub-Committee XXI, Committee D-2, 290.
  Petroleum Products and Lubricants. Report of Committee D-2, 248.
  Proposed Method for Determination of Oil in Wax. Report of Sub-Committee III, Committee D-2, Appendix, 263.
  Proposed Method of Test for Neutralization Number of Petroleum Oil, Titration by Electrometric Method. Report of Sub-Committee XIII, Committee D-2, Appendix, 282.

Petroleum Products (*Continued*):
  Results of investigation on modified A.S.T.M. steam emulsion test, 273.
  Results of sulfur determinations of motor fuels containing benzol, 269.
  Results of tests on determination of oil in wax of varying oil content, 261.
  Results of tests to determine naphtha content of crude oils, 291.
  Tentative Method of Test for Cloud and Pour Points of Petroleum Products, 725.
  Tentative Method of Test for Distillation of Gasoline, Naptha, Kerosine, and Similar Petroleum Products, 685.
  Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants, 722.
  Tentative Methods of Test for Viscosity of Petroleum Products and Lubricants, 707.
Pigments.
  Tentative Specifications for Bone Black, 652.
  Tentative Specifications for Chrome Yellow, 654.
  Tentative Specifications for Iron Oxide and Iron Hydroxide, 646.
  Tentative Specifications for Lampblack, 650.
  Tentative Specifications for Lithopone, 648.
  Tentative Specifications for Pure Chrome Green, 656.
  Tentative Specifications for Reduced Chrome Green, 658.
Pipe.
  Tentative Revision of Standard Specifications for Cement-Concrete Sewer Pipe, 904.
  Tentative Revision of Standard Specifications for Welded and Seamless Steel Pipe, 896.
  Tentative Revision of Standard Specifications for Welded Wrought-Iron Pipe, 897.
Pothead Compounds.
  Tentative Methods of Testing Cable Splicing and Pothead Compounds, 810.
Pour Point.
  Proposed Specifications for A.S.T.M. Low-Cloud and Pour Test Thermometers, −60 to +20° C., −70 to +70° F. Report of Committee D-15, 393.
  Tentative Method of Test for Cloud and Pour Points of Petroleum Products, 725.
Preservatives.
  Preservative Coatings for Structural Materials. Committee D-1, 220.
  Timber Preservatives. Report of Sub-Committee VI, Committee D-7, 340.
Publications.
  Papers and Publications. Report of Committee E-6, 486.
Pure Chrome Green.
  Tentative Specifications for Pure Chrome Green, 656.

Q

Quicklime.
  See **Lime.**

R

Radio Frequencies.
  Tentative Method of Testing Electrical Insulating Materials for Voltage Effects at Radio Frequencies, 804.
Reduced Chrome Green.
  Tentative Specifications for Reduced Chrome Green, 658.

Refractories.
Classification of refractories for by-product coke ovens, 196.
Classification of refractories for malleable-cast-iron industry, 195.
Refractories. Report of Committee C-8, 190.
Report of Industrial Survey. Report of Committee C-8, Appendix, 192.

Research.
Correlation of Research. Report of Committee E-9, 502.
Promotion of Knowledge of Engineering Materials. Annual Address by the President, F. M. Farmer, 34.

Road Materials.
Road and Paving Materials. Report of Committee D-4, 293.
Tentative Method of Float Test for Bituminous Materials, 748.
Tentative Method of Test for Consistency of Portland-Cement Concrete, 746.
Tentative Methods of Mechanical Analysis of Subgrade Soils, 742.
Tentative Methods of Test for the Determination of Moisture Equivalent of Subgrade Soils in the Field, 745.
Tentative Revision of Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment, 911.
Tentative Revision of Standard Specifications for Broken Slag for Waterbound Base and Wearing Course, 910.
Tentative Revision of Standard Specifications for Shovel-Run or Crusher Run Broken Slag for Waterbound Base, 910.
Tentative Specifications for High-Carbon Tar Cement, 740.
Tentative Specifications for High-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product), 738.

Roll-Roofing.
Tentative Methods of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles, 792.
Tentative Specifications for Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles, 765.
Tentative Specifications for Smooth-Surfaced Asphalt Roll-Roofing, 761.

Roofing.
Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 342.
Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing, 776.
Tentative Methods of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles,. 792.
Tentative Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs, 770.
Tentative Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs, 773.
Tentative Specifications for Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles, 765.
Tentative Specifications for Smooth-Surfaced Asphalt Roll-Roofing, 761.

Rubber Products.
Performance tests of rubber products, 374.
Proposed specifications for rubber disks for alarm valves in sprinkler systems, 372.
Rubber Products. Report of Committee D-11, 371.

S

Sand.
Tentative Definition of the Term Sand, 635.

Saponification.
Neutralization Number and Saponification. Report of Sub-Committee XIII, Committee D-2, 280.

Scoured Content.
Tentative Methods of Testing Grease Wool and Allied Fibers for Scoured Content, 846.

Screen.
Screen Wire Cloth. Report of Committee D-14, 384.
Tentative Definition of the Term Screen (Sieve), 889.
Tentative Specifications for Non-Ferrous Insect Screen Cloth, 587.
Tentative Specifications for Sieves for Testing Purposes, 850.

Sewer Pipe.
Tentative Revision of Standard Specifications for Cement-Concrete Sewer Pipe, 904.

Shear.
Proposed Methods for Torsion Tests to Determine the Mechanical Properties of Metallic Materials Under Shearing Stress. Report of Committee E-1, Appendix II, 430.

Sheet.
Tentative Methods of Testing Laminated Sheet Insulating Materials, 825.
Tentative Revision of Standard Specifications for High Sheet Brass, 903.

Shellac.
Tentative Methods of Testing Shellac Varnish, 660.
Tentative Specifications for Dry Bleached Shellac, 644.

Shingles.
Tentative Methods of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing, and Slate-Surfaced Asphalt Shingles, 792.
Tentative Specifications for Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles, 765.

Sieve.
Tentative Definition of the Term Screen (Sieve), 889.
Tentative Specifications for Sieves for Testing Purposes, 850.

Silicon.
Tentative Specifications for Ferro-Silicon, 531.
Tentative Specifications for Structural Silicon Steel, 514.

Slag.
Tentative Revision of Standard Specifications for Broken Slag for Waterbound Base and Wearing Course, 910.
Tentative Revision of Standard Specifications for Shovel-Run or Crusher-Run Broken Slag for Waterbound Base, 910.

Slate.
Slate. Report of Committee D-16, 395.
Tentative Methods of Flexure Testing of Slate (Determination of Modulus of Rupture and Modulus of Elasticity), 756.
Tentative Method of Test for Water Absorption of Slate, 754.

Slump Test.
Tentative Method of Test for Consistency of Portland-Cement Concrete, 746.

Soil.
Tentative Method of Mechanical Analysis of Subgrade Soils, 742.
Tentative Method of Test for the Determination of Moisture Equivalent of Subgrade Soils in the Field, 745.

Southern Pine.
See **Timber.**

Specific Gravity.
Tentative Definitions of Terms Relating to Specific Gravity, 887.

Spiegeleisen.
Tentative Specifications for Spiegeleisen, 529.

Standardization.
Promotion of Knowledge of Engineering Materials. Annual Address by the President, F. M. Farmer, 34.

Staybolts.
Tentative Specifications for Hollow Staybolt Iron, 534.
Tentative Revision of Standard Specifications for Staybolt, Engine-Bolt and Extra-Refined Wrought-Iron Bars, 897.

Steel.
Corrosion of Iron and Steel. Report of Committee A-5, 95.
Heat Treatment of Iron and Steel. Report of Committee A-4, 94.
Inspection of the Fort Sheridan, Pittsburgh, and Annapolis Tests. Report of Sub-Committee III, Committee A-5, 97.
Outline of atmospheric exposure tests on zinc-coated products, 104.
Preparation of Iron and Steel Surfaces for Painting. Report of Sub-Committee XIV, Committee D-1, 238.
Proposed Revisions in Standards for Steel. Report of Committee A-1, Appendix, 75.
Steel. Report of Committee A-1, 70.
Tentative Recommended Practice for Carburizing and Heat-Treatment of Carburized Objects, 568.
Tentative Recommended Practice for Thermal Analysis of Steel, 890.
Tentative Specifications for Carbon-Steel Car and Tender Axles, 509.
Tentative Specifications for Carbon-Steel Castings for Valves, Flanges and Fittings for High-Temperature Service, 519.
Tentative Specifications for Structural Silicon Steel, 514.
Tentative Revision of Standard Recommended Practice for Heat Treatment of Case-Hardened Carbon-Steel Objects, 898.
Tentative Revision of Standard Specifications for Carbon-Steel and Alloy-Steel Forgings, 895.
Tentative Revision of Standard Specifications for Carbon-Steel Car and Tender Axles, 896.
Tentative Revision of Standard Specifications for Carbon-Steel Forgings for Locomotives, 895.
Tentative Revision of Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes, 896.
Tentative Revision of Standard Specifications for Quenched-and-Tempered Alloy-Steel Axles, Shafts and Other Forgings for Locomotives and Cars, 895.

Steel (*Continued*):

Tentative Revision of Standard Specifications for Quenched-and-Tempered Carbon-Steel Axles, Shafts and Other Forgings for Locomotives and Cars, 895.

Tentative Revision of Standard Specifications for Welded and Seamless Steel Pipe, 896.

Total Immersion Tests. Report of Sub-Committee V, Committee A-5, 99.

Subgrade.

Tentative Method of Mechanical Analysis of Subgrade Soils, 742.

Tentative Method of Test for the Determination of Moisture Equivalent of Subgrade Soils in the Field, 745.

Sulfur.

Report on collaborative tests on detection of sulfur in mineral spirits, 222.

Results of sulfur determinations of motor fuels containing benzol, 269.

Sulfur Determination and Differentiation. Report of Sub-Committee VII, Committee D-2, 269.

Summary of the Proceedings.

Summary of the Proceedings of the Twenty-eighth Annual Meeting, 11.

T

Tar.

Tentative Method of Float Test for Bituminous Materials, 748.

Tentative Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs, 773.

Tentative Specifications for High-Carbon Tar Cement, 740.

Tentative Specifications for High-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product), 738.

Tension Testing.

Digest of Literature on Methods of Making Flexure and Tension Tests of Concrete by Duff A. Abrams. Report of Committee C-9, Appendix I, 205.

Tentative Methods of Tension Testing of Metallic Materials, 854.

Tentative Specifications for Textile Testing Machines, 829.

Testing Machines.

Tentative Specifications for Textile Testing Machines, 829.

Testing, Methods of.

Methods of Testing. Report of Committee E-1, 398. Discussion, 437.

Tentative Definitions of Terms Relating to Methods of Testing, 879.

Textile Materials.

Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing, 776.

Tentative Methods of Testing Grease Wool and Allied Fibers for Scoured Content, 846.

Tentative Specifications for Textile Testing Machines, 829.

Tentative Specifications for Tolerances for Numbered Cotton Duck, 840.

Tentative Specifications for Tolerances and Test Methods for Cotton Sewing Threads, 837.

Tentative Specifications for Tolerances and Test Methods for Cotton Yarns, Single and Plied, 831.

Tentative Specifications for Tolerances and Test Methods for Knit Goods, 842.

Textile Materials. Report of Committee D-13, 376.

Thermal Analysis.
Tentative Recommended Practice for Thermal Analysis of Steel, 890.

Thermometers.
Proposed Specifications for A.S.T.M. Low-Cloud and Pour Test Thermometers, −60 to +20° C., −70 to +70° F. Report of Committee D-15, 393.
Specifications for cloud and pour test thermometer, Tentative Method of Test for Cloud and Pour Points of Petroleum Products, 725.
Specifications for high-distillation thermometer, Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products, 689.
Specifications for low cloud and pour test thermometer, Tentative Method of Test for Cloud and Pour Points of Petroleum Products, 726.
Specifications for low-distillation thermometer, Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products, 688; also, Tentative Method of Test for Distillation of Natural Gas Gasoline, 696.
Specifications for low-softening point thermometer, Tentative Method of Float Test for Bituminous Materials, 748.
Specifications for thermometers used in viscosity tests, Tentative Methods of Test for Viscosity of Petroleum Products and Lubricants, 709.
Thermometers. Report of Committee D-15, 391.

Threads.
Tentative Specifications for Tolerances and Test Methods for Cotton Sewing Threads, 837.

Tile.
Hollow Masonry Building Units. Report of Committee C-10, 210.

Timber.
Specifications for Timber. Report of Sub-Committee I, Committee D-7, 312.
Timber. Report of Committee D-7, 310.
Timber Preservatives. Report of Sub-Committee VI, Committee D-7, 340.
Use Specifications (A): Buildings, 333.
Use Specifications (B): Bridges and Trestles, 334.
Working Stresses for Douglas Fir and Southern Pine. Report of Sub-Committee I, Committee D-7, Appendix, 335.

Torsion.
Proposed Methods for Torsion Tests to Determine the Mechanical Properties of Metallic Materials Under Shearing Stress. Report of Committee E-1, Appendix II, 430.

Trolley Wire.
Tentative Revision of Standard Specifications for High Strength Bronze Trolley Wire, Round and Grooved, 40 and 65-per-cent Conductivity, 902.

Tubes.
Tentative Specifications for Muntz Metal Condenser Tube Plates, 583.
Tentative Revision of Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes, 896.
Tentative Revision of Standard Specifications for Seamless Admiralty Condenser Tubes and Ferrule Stock, 902.

Tung Oil.
Tentative Specifications for Raw Tung Oil, 636.

Tungsten.
Tentative Specifications for Tungsten Powder, 528.
Turpentine.
Tentative Revision of Standard Specifications for Turpentine, 909.

V

Valves.
Tentative Specifications for Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service, 519.
Varnish.
Tentative Methods of Testing Insulating Varnishes, 797.
Tentative Methods of Testing Shellac Varnish, 660.
Varnish. Report of Sub-Committee IX, Committee D-1, 233.
Viscosity.
Tentative Methods of Test for Viscosity of Petroleum Products and Lubricants, 707.
Tentative Methods of Testing Gas Oils (Gravity, Distillation, Sulfur, Carbon Residue, Pour Point, Viscosity, Water), 732.
Voltage Effects.
Tentative Method of Testing Electrical Insulating Materials for Voltage Effects at Radio Frequencies, 804.

W

Water.
Application of distillation methods for moisture, 421.
Determination of Water. Report of Sub-Committee, Committee E-1, 416.
Summary of various methods for determination of moisture, 419.
Water Treatment.
Tentative Specifications for Hydrated Lime for Use in Water Treatment, 616.
Tentative Specifications for Quicklime for Use in Water Treatment, 614.
Waterproofing.
Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 342.
Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures, 786.
Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing, 776.
Tentative Methods of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles, 792.
Tentative Specifications for Acid-Resisting Asphalt Mastic, 759.
Tentative Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs, 770.
Tentative Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs, 773.
Tentative Specifications for Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles, 765.
Tentative Specifications for Smooth-Surfaced Asphalt Roll-Roofing, 761.
Wax.
Paraffin Wax. Report of Sub-Committee III, Committee D-2, 260.
Proposed Method for Determination of Oil in Wax. Report of Sub-Committee III, Committee D-2, Appendix, 263.
Results of tests on determination of oil in wax of varying oil content, 261.

White Metals.
Review of the Antimony Situation by H. K. Masters. Report of Committee B-2, Appendix, 143.

Wire.
Copper Wire. Report of Committee B-1, 126.
Proposed Requirements for Zinc-Coated or Galvanized Wire. Report of Sub-Committee VI, Committee A-5, Appendix, 102.
Screen Wire Cloth. Report of Committee D-14, 384.
Tentative Revision of Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars, 901.
Tentative Revision of Standard Specifications for High-Strength Bronze Trolley Wire, Round and Grooved, 40 and 65-per-cent Conductivity, 902.
Tentative Revision of Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars, 898.
Tentative Specifications for Bronze Trolley Wire, 578.
Tentative Specifications for Non-Ferrous Insect Screen Cloth, 587.

Wool.
Tentative Methods of Testing Grease Wool and Allied Fibers for Scoured Content, 846.

Wrought Iron.
Proposed methods of sampling and chemical analysis of wrought iron, 79.
Results of vibratory and tension tests on staybolt iron, annealed and unannealed, 82.
Tentative Specifications for Hollow Staybolt Iron, 534.
Tentative Revision of Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes, 896.
Tentative Revison of Standard Specifications for Staybolt, Engine-Bolt and Extra-Refined Wrought-Iron Bars, 897.
Tentative Revision of Standard Specifications for Welded Wrought-Iron Pipe, 897.
Wrought Iron. Report of Committee A-2, 77.

X

X-ray.
Bibliography on metal radiography and X-ray crystallography, 467.
Discussion on X-ray metallography, 470.
Metal radiography, 444.
X-ray crystallography, 448.
X-ray Metallography. Report of Sub-Committee VI, Committee E-4, 444.

Y

Yarns.
Tentative Specifications for Tolerances and Test Methods for Cotton Yarns, Single and Plied, 831.

Z

Zinc.
Outline of atmospheric exposure tests on zinc-coated products, 104.
Proposed Requirements for Zinc-Coated or Galvanized Wire. Report of Sub-Committee VI, Committee A-5, Appendix, 102.

AUTHOR INDEX

A

Abrams, D. A.
Report of Committee C-1, Laboratory Manual of Cement Testing, Appendix: Brief Bibliography on Portland Cement, 172.
Report of Committee C-9, Appendix I: Digest of Literature on Methods of Making Flexure and Tension Tests of Concrete, 205.

Aston, James
Report of Committee A-5 on Corrosion of Iron and Steel, 95.

B

Bassett, W. H.
Report of Committee D-14 on Screen Wire Cloth, 384.

Berry, H. C.
Report of Committee C-7 on Lime, 186.

Bowles, J. T-B.
Report of Sub-Committee XI, Committee D-2, on Emulsification, 272.

Boyd, D. K.
Report of Committee D-16 on Slate, 395.

Boylston, H. M.
Report of Committee A-4 on Heat Treatment of Iron and Steel, 94.

Boynton, H. C.
Report of Committee E-4 on Metallography, 440.

Brown, R. P.
Report of Committee C-7 on Lime, 186.

Butterfield, E. E.
Discussion, 181.

C

Campbell, W.
Report of Committee B-2 on Non-Ferrous Metals and Alloys, 129.
Addendum to Report of Committee B-2, 149.

Capp, J. A.
Report of Committee B-1 on Copper Wire, 126.
Report of Committee E-1 on Methods of Testing, 398.

Carpenter, A. W.
Report of Sub-Committee XIV, Committee D-1, on Preparation of Iron and Steel Surfaces for Painting, 238.

Chapman, C. M.
Report of Committee E-8 on Nomenclature and Definitions, 488.

Church, S. R.
Report of Sub-Committee VI, Committee D-7, on Timber Preservatives, 340.

Cole, E. J.
Report of Sub-Committee IX, Committee D-1, on Varnish, 233.

Comstock, G. F.
  Report of Committee E-4 on Metallography, 440.
  Discussion, 437.

Cook, K. B.
  Report of Committee D-13 on Textile Materials, 376.

Cooper, W. A.
  Report of Sub-Committee V, Committee A-5, on Total Immersion Tests, 99.

Cox, D. C.
  Report of Committee D-9, Appendix: Life Test for Transformer Oils, 363.

D

Davies, G. C.
  Report of Committee A-3 on Cast Iron, 84.

E

Edwards, E. J.
  Discussion, 88.

Edwards, W. F.
  Report of Committee D-13 on Textile Materials, 376.

Emley, W. E.
  Report of Committee C-3 on Brick, 184.
  Report of Committee C-11 on Gypsum, 211.
  Discussion, 218.

F

Farmer, F. M.
  Annual Report of the Executive Committee, 46.
  Discussion, 369.
  Promotion of Knowledge of Engineering Materials. Annual Address by the President, F. M. Farmer, 34.
  Report of Committee D-9 on Electrical Insulating Materials, 348.
  Report of Committee D-11 on Rubber Products, 371.

Fieldner, A. C.
  Report of Committee D-5 on Coal and Coke, 307.

Flagg, S. G., Jr.
  Discussion, 87.

Force, H. J.
  Report of Committee A-2 on Wrought Iron, 77.

Forrest, C. N.
  Report of Sub-Committee XV, Committee D-2, on Sampling and Gaging, 286.

Fulweiler, W. H.
  Report of Committee C-8 on Refractories, 190.
  Report of Committee D-15 on Thermometers, 391.
  Report of Sub-Committee, Committee E-1, on Determination of Water, 416.

G

Gardner, H. A.
  Report of Sub-Committee III, Committee D-1, on Testing Paint Vehicles, 227.

Gibboney, J. H.
Report of Committee A-5 on Corrosion of Iron and Steel, 95.
Report of Sub-Committee III, Committee A-5, on Inspection of the Fort Sheridan, Pittsburgh, and Annapolis Tests, 97.
Discussion, 89, 120.

Gilbert, H. W.
Discussion, 119.

Goldbeck, A. T.
Report of Committee C-9 on Concrete and Concrete Aggregates, 199.

Grafton, E. H.
Report of Committee D-11 on Rubber Products, 371.

Greenman, R. S.
Report of Committee C-1 on Cement, 159.

H

Hall, J. H.
Report of Committee A-4 on Heat Treatment of Iron and Steel, 94.

Hallett, R. L.
Report of Committee D-1 on Preservative Coatings for Structural Materials, 220.

Harvey, F. A.
Report of Committee C-8 on Refractories, 190.

Hecht, Max.
Report of Sub-Committee XIII, Committee D-2, on Neutralization Number and Saponification, 280.

Herschel, W. H.
Discussion, 369.

Hoffman, N. B.
Discussion, 123.

Hubbard, Prévost.
Report of Committee D-4 on Road and Paving Materials, 293.
Report of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 342.

Hull, F. A.
Report of Sub-Committee VIII, Committee A-5, on Tests of Metallic-Coated Products, 104.
Report of Sub-Committee XXIV, Committee D-2, on Calorific Value of Fuel Oil, 292.

Huse, F. J.
Report of Committee C-10 on Hollow Masonry Building Units, 210.

I

Ingalls, F. P.
Report of Sub-Committee XVIII, Committee D-1, on Physical Properties of Paint Materials, 241.

Ingberg, S. H.
Report of Committee C-10 on Hollow Masonry Building Units, 210.
Discussion, 218.

J

Jasper, T. McL.
Discussion, 485.

Jeffries, Zay.
Report of Sub-Committee VI, Committee E-4, on X-ray Metallography, 444.

Johansen, E. M.
Report of Sub-Committee VII, Committee D-2, on Sulfur Determination and Differentiation, 269.

Johnson, N. C.
Discussion, 180.

K

Kessler, D. W.
Report of Committee D-16 on Slate, 395.

L

Langenberg, F. C.
Report of Committee A-9 on Ferro-Alloys, 121.

Lathrop, E. C.
Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, 150.

Law, L. M.
Report of Committee D-4, Appendix: Report of Sectional Committee on Penetration, 301.

Lawson, T. R.
Report of Committee C-3 on Brick, 184.

M

MacKenzie, J. T.
Discussion, 89, 123.

Mackenzie, K. G.
Report of Sub-Committee XX, Committee D-2, on Nomenclature, 287.

Malcolm, V. T.
Discussion, 470.

Manning, Van. H.
Report of Committee D-2 on Petroleum Products and Lubricants, 248.

Marani, V. G.
Report of Committee C-11 on Gypsum, 211.
Discussion, 219.

Masters, H. K.
Report of Committee B-2, Appendix: Review of the Antimony Situation, 143.

Mattimore, H. S.
Report of Committee D-4 on Road and Paving Materials, 293.

McKnight, Charles, Jr.
Report of Committee A-9 on Ferro-Alloys, 121.

Merica, P. D.
Report of Committee B-2 on Non-Ferrous Metals and Alloys, 129.
Addendum to Report of Committee B-2, 149.

Merriman, Thaddeus.
Discussion, 178.

Moldenke, Richard.
Report of Committee A-3 on Cast Iron, 84.
Discussion, 87

Moore, H. F.
Discussion, 438.
Report of Committee E-9 on Correlation of Research, 502.

Muckenfuss, A. M.
Report of Sub-Committee XXIII, Committee D-1, on Anti-Fouling Paints, 244.

Mueller, E. F.
Report of Committee D-15 on Thermometers, 391.

Nemzek, L. P.
Report of Sub-Committee VI, Committee D-1, on Definitions of Terms Used in Paint Specifications, 232.

N

Newlin, J. A.
Report of Committee D-7 on Timber, 310.

O

Osterhout, C. C.
Report of Committee A-2 on Wrought Iron, 77.

P

Paul, C. E.
Report of Sub-Committee I, Committee D-7, on Specifications for Timber, 312.

Pearson, J. C.
Report of Committee C-9 on Concrete and Concrete Aggregates, 199.

Phillips, E. B.
Report of Sub-Committee III, Committee D-2, on Paraffin Wax, 260.

Porter, H. C.
Report of Committee D-5 on Coal and Coke, 307.

Putnam, W. P.
Report of Committee A-7 on Malleable Castings, 118.

R

Rawdon, H. S.
Report of Sub-Committee VII, Committee A-5, on Accelerated Tests, 103.
Report of Sub-Committee V, Committee E-4, on Micro-Hardness, 442.

Rogers, Allen.
Report of Committee D-1 on Preservative Coatings for Structural Materials, 220.

Rogers, R. E.
Report of Sub-Committee XXII, Committee D-1, on Method of Application of Paint by Spraying, 243.

S

St. John, Ancel.
Discussion, 484.

Selvig, W. A.
Report of Committee D-5 on Coal and Coke, 307.

Slater, W. A.
Discussion, 181.

Smith, H. E.
Report of Sub-Committee VI, Committee A-5, on Specifications for Metallic-Coated Products, 101.
Smith, N. A. C.
Report of Sub-Committee XXI, Committee D-2, on Crude Petroleum, 290.
Snyder, E. A.
Report of Committee D-9, Appendix: Life Test for Transformer Oils, 363.
Spring, L. W.
Discussion, 124.

T

Touceda, Enrique.
Report of Committee A-7 on Malleable Castings, 118.
Tour, Sam.
Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, 150.

V

Van Gundy, C. P.
Report of Committee D-2 on Petroleum Products and Lubricants, 248.
Vassar, H. S.
Report of Committee D-9 on Electrical Insulating Materials, 348.
von Schrenk, Hermann.
Report of Committee D-7 on Timber, 310.

W

Wagner, S. T.
Report of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 342.
Waring, F. M.
Report of Committee A-1 on Steel, 70.
Warwick, C. L.
Annual Report of the Executive Committee, 46.
Discussion, 89.
Report of Committee E-1 on Methods of Testing, 398.
Report of Committee E-6 on Papers and Publications, 486.
Report of Committee E-8 on Nomenclature and Definitions, 488.
Report of Committee E-9 on Correlation of Research, 502.
Wiggin, T. H.
Discussion, 89.
Wilson, R. E.
Report of Sub-Committee IV, Committee D-2, on Grease, 266.
Woodroffe, G. H.
Report of Committee A-1 on Steel, 70.
Woodward, R. W.
Report of Committee D-14 on Screen Wire Cloth, 384.
Discussion, 438.

Y

Young, C. D.
Discussion, 124.
Young, R. B.
Report of Committee C-1 on Cement, 159.

LIST OF STANDARDS[1]

Published Collectively in their latest revised form in 1924 BOOK OF A.S.T.M. STANDARDS with 1925 Supplement to the BOOK OF STANDARDS

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A. FERROUS METALS

STEEL

See also **WROUGHT IRON:** A 56 – 24

STANDARD SPECIFICATIONS

Steel Rails and Accessories.

A 1 – 24. For Carbon-Steel Rails.
A 2 – 24. For the Manufacture of Open-hearth Steel Girder Rails of Plain, Grooved and Guard Types.
A 3 – 24. For Low-Carbon-Steel Splice Bars.
A 4 – 14. For Medium-Carbon-Steel Splice Bars.
A 5 – 14. For High-Carbon-Steel Splice Bars.
A 6 – 14. For Extra-High-Carbon-Steel Splice Bars.
A 49 – 21. For Quenched High-Carbon-Steel Splice Bars.
A 50 – 24. For Quenched Carbon-Steel Track Bolts.
A 51 – 21. For Quenched Alloy-Steel Track Bolts.
A 65 – 24. For Steel Track Spikes.
A 66 – 21. For Steel Screw Spikes.

Structural Steel.

A 7 – 24. For Structural Steel for Bridges.
A 8 – 24. For Structural Nickel Steel.
A 9 – 24. For Structural Steel for Buildings.
A 10 – 24. For Structural Steel for Locomotives.
A 11 – 24. For Structural Steel for Cars.
A 12 – 21. For Structural Steel for Ships.
A 13 – 24. For Rivet Steel for Ships.

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[1] As of August 28, 1925. Total number, 254.

Spring Steel and Springs.

A 14 - 16. For Carbon Steel-Bars for Railway Springs.
A 68 - 18. For Carbon-Steel Bars for Railway Springs with Special Silicon Requirements.
A 58 - 16. For Carbon-Steel Bars for Vehicle and Automobile Springs.
A 59 - 16. For Silico-Manganese-Steel Bars for Automobile and Railway Springs
A 60 - 16. For Chrome-Vanadium-Steel Bars for Automobile and Railway Springs.
A 61 - 16. For Helical Steel Springs for Railways.
A 62 - 16. For Elliptical Steel Springs for Railways.
A 69 - 18. For Elliptical Steel Springs for Automobiles.

Reinforcement Bars.

A 15 - 14. For Billet-Steel Concrete Reinforcement Bars.
A 16 - 14. For Rail-Steel Concrete Reinforcement Bars.

Steel Blooms, Forgings and Axles.

*A 17 - 21. For Carbon-Steel and Alloy-Steel Blooms, Billets and Slabs for Forgings.
*A 18 - 21. For Carbon-Steel and Alloy-Steel Forgings.
A 19 - 21. For Quenched-and-Tempered Carbon-Steel Axles, Shafts and Other Forgings for Locomotives and Cars.
A 63 - 21. For Quenched-and-Tempered Alloy-Steel Axles, Shafts, and Other Forgings for Locomotives and Cars.
A 20 - 21. For Carbon-Steel Forgings for Locomotives.
A 21 - 18. For Carbon-Steel Car and Tender Axles.
A 22 - 21. For Cold-Rolled Steel Axles.

Steel Wheels and Tires.

A 25 - 24. For Wrought Solid Carbon-Steel Wheels for Electric Railway Service.
A 57 - 24. For Wrought Solid Carbon-Steel Wheels for Steam Railway Service.
A 26 - 16. For Steel Tires.

Steel Castings.

A 27 - 24. For Steel Castings.

Steel Tubes and Pipe.

A 83 - 24. For Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes.
A 53 - 24. For Welded and Seamless Steel Pipes.

Automotive Steels (see also **Spring Steel**).

A 29 - 24. For Automotive Carbon and Alloy Steels.

Boiler Steels.

A 30 - 24. For Boiler and Firebox Steel for Locomotives.
A 70 - 24. For Boiler and Firebox Steel for Stationary Service.
A 31 - 24. For Boiler Rivet Steel.

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Bar Steels.

A 32 – 24. For Cold-Finished Bessemer Steel Automatic Screw Stock.
A 54 – 24. For Cold-Finished Open-Hearth Steel Automatic Screw Stock.
A 80 – 24. For Commercial Bar Steels.

STANDARD METHODS

A 34 – 24. Of Test for Magnetic Properties of Iron and Steel.
A 33 – 24. Of Chemical Analysis of Plain Carbon Steel.
A 55 – 24. Of Chemical Analysis of Alloy Steels.
A 90 – 24. Of Determining Weight of Coating on Zinc-Coated Articles.
A 91 – 24. Of Determining Weight of Coating on Tin, Terne, and Lead-Coated Sheets.

RECOMMENDED PRACTICE

A 35 – 24. For Annealing of Miscellaneous Rolled and Forged Carbon-Steel Objects.
A 36 – 24. For Heat Treatment of Carbon-Steel Castings.
A 37 – 14. For Heat Treatment of Case-Hardened Carbon-Steel Objects.

WROUGHT IRON

(See also **STEEL:** A 83 – 24)

STANDARD SPECIFICATIONS

A 72 – 24. For Welded Wrought-Iron Pipe.
A 84 – 24. For Staybolt, Engine-Bolt and Extra-Refined Wrought-Iron Bars.
*A 41 – 18. For Refined Wrought-Iron Bars.
A 85 – 24. For Merchant Bar Iron.
*A 42 – 18. For Wrought-Iron Plates.
A 73 – 24. For Wrought-Iron Rolled or Forged Blooms and Forgings for Locomotives and Cars.
A 56 – 24. For Iron and Steel Chain.

STANDARD DEFINITIONS

A 81 – 21. Of Terms Relating to Wrought-Iron Specifications.

PIG IRON, CAST IRON, AND FINISHED CASTINGS

STANDARD SPECIFICATIONS

A 43 – 24. For Foundry Pig Iron.
A 44 – 04. For Cast-Iron Pipe and Special Castings.
A 74 – 18. For Cast-Iron Soil Pipe and Fittings.
A 45 – 14. For Cast-Iron Locomotive Cylinders.
A 46 – 24. For Chilled Cast-Iron Wheels.
A 47 – 24. For Malleable Castings.
A 48 – 18. For Gray-Iron Castings.
A 88 – 24. For High-Test Gray-Iron Castings.

STANDARD METHODS

A 64 – 16. Of Sampling and Chemical Analysis of Pig and Cast Iron.

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B. NON-FERROUS METALS

STANDARD SPECIFICATIONS

Ingot Copper.

*B 4 - 13. For Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars.
*B 5 - 13. For Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars.

Spelter.

B 6 - 18. For Spelter.

Lead.

B 29 - 23. For Pig Lead.

Nickel.

B 39 - 22. For Nickel.

Aluminum Alloys.

B 26 - 21. For Light Aluminum Casting Alloys.

Wire and Cable.

B 1 - 23. For Hard-Drawn Copper Wire.
B 2 - 15. For Medium Hard-Drawn Copper Wire.
*B 3 - 15. For Soft or Annealed Copper Wire.
B 33 - 21. For Tinned Soft or Annealed Copper Wire for Rubber Insulation.
B 8 - 21. For Bare Concentric-Lay Copper Cable: Hard, Medium-Hard, or Soft.
B 47 - 25. For Round and Grooved Hard-Drawn Copper Trolley Wire.
B 9 - 16. For High-Strength Bronze Trolley Wire, Round and Grooved: 40 and 65-per-cent Conductivity.

Bronze.

B 10 - 18. For the Alloy: Copper, 88 per cent; Tin, 10 per cent; Zinc, 2 per cent.
*B 30 - 22. For Brass Ingot Metal, Graded and Ungraded, for Sand Castings.
B 31 - 21. For Bronze Bearing Metal in Ingot Form.
B 22 - 21. For Bronze Bearing Metals for Turntables and Movable Railroad Bridges.

Solder Metal.

*B 32 - 21. For Solder Metal.

Copper and Brass Plates, Tubes, Rods, etc.

B 11 - 18. For Copper Plates for Locomotive Fireboxes.
B 12 - 21. For Copper Bars for Locomotives Staybolts.
B 13 - 18. For Seamless Copper Boiler Tubes.
B 14 - 18. For Seamless Brass Boiler Tubes.
B 44 - 24. For Seamless Admiralty Condenser Tubes and Ferrule Stock.
B 55 - 25. For Seamless 70–30 Brass Condenser Tubes and Ferrule Stock.
B 56 - 25. For Seamless Muntz Metal Condenser Tubes and Ferrule Stock.
B 42 - 24. For Copper Pipe, Standard Sizes.

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Copper and Brass Plates, Tubes, Rods, etc.—Continued.

B 43 - 24.	For Brass Pipe, Standard Sizes.
*B 15 - 18.	For Brass Forging Rod.
*B 16 - 18.	For Free-Cutting Brass Rod for Use in Screw Machines.
B 19 - 19.	For Cartridge Brass.
B 20 - 19.	For Cartridge Brass Disks.
*B 21 - 19.	For Naval Brass Rods for Structural Purposes.
*B 36 - 21.	For High Sheet Brass.

STANDARD METHODS

*B 34 - 20.	Of Battery Assay of Copper.
B 35 - 24.	Of Chemical Analysis of Pig Lead.
B 38 - 21.	Of Chemical Analysis of Spelter.
B 41 - 23.	Of Chemical Analysis of Nickel.
*B 18 - 21.	Of Chemical Analysis of Alloys of Lead, Tin, Antimony and Copper.
*B 27 - 19.	Of Chemical Analysis of Manganese Bronze
*B 28 - 19.	Of Chemical Analysis of Gun Metal.
B 45 - 23.	Of Chemical Analysis of Brass Ingots and Sand Castings.
B 46 - 23.	Of Chemical Analysis of Bronze Bearing Metal.

C. CEMENT, LIME, GYPSUM, AND CLAY PRODUCTS

STANDARD SPECIFICATIONS

†C 9 - 21.	Specifications and Tests for Portland Cement.
C 10 - 09.	For Natural Cement.
C 7 - 15.	For Paving Brick.
C 21 - 20.	For Building Brick.
C 32 - 24.	For Clay Sewer Brick.
C 13 - 24.	For Clay Sewer Pipe.
C 14 - 24.	For Cement-Concrete Sewer Pipe.
C 4 - 24.	For Drain Tile.
C 6 - 24.	For Hydrated Lime for Structural Purposes.
C 45 - 25.	For Quicklime and Hydrated Lime for Use in the Cooking of Rags for the Manufacture of Paper.
C 48 - 24.	For Quicklime and Hydrated Lime for Use in the Textile Industry.
C 49 - 24.	For Quicklime and Hydrated Lime for the Manufacture of Silica Brick.
C 22 - 25.	For Gypsum.
C 23 - 22.	For Calcined Gypsum.
C 28 - 21.	For Gypsum Plasters.
C 35 - 25.	For Gypsum Plastering Sand.
C 36 - 25.	For Gypsum Wall Board.
C 37 - 25.	For Gypsum Plaster Board.
C 52 - 25.	For Gypsum Partition Tile or Block.
*C 19 - 18.	For Fire Tests of Materials and Construction.

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STANDARD METHODS

C 16 - 20.	Of Test for Refractory Materials under Load at High Temperatures.
C 20 - 20.	Of Test for Porosity and Permanent Volume Changes in Refractory Materials.
C 24 - 20.	Of Test for Softening Point of Fire-Clay Brick.
C 18 - 21.	Of Ultimate Chemical Analysis of Refractory Materials, Including Chrome Ores and Chrome Brick.
*C 29 - 21.	Of Test for Unit Weight of Aggregate for Concrete.
*C 30 - 22.	Of Test for Voids in Fine Aggregate for Concrete.
C 31 - 21.	Of Making and Storing Specimens of Concrete in the Field.
C 39 - 25.	Of Making Compression Tests of Concrete.
*C 40 - 22.	Of Test for Organic Impurities in Sands for Concrete.
C 41 - 24.	Of Test for Sieve Analysis of Aggregates for Concrete.
C 26 - 23.	Of Testing Gypsum and Gypsum Products.

STANDARD DEFINITIONS

C 8 - 24.	Of Terms Relating to Sewer Pipe.
C 27 - 20.	For Clay Refractories.
C 43 - 24.	Of Terms Relating to Hollow Tile.

RECOMMENDED PRACTICE

C 12 - 19.	For Laying Sewer Pipe.

D. MISCELLANEOUS MATERIALS

PRESERVATIVE COATINGS

STANDARD SPECIFICATIONS

D 1 - 15.	For Purity of Raw Linseed Oil from North American Seed.
D 11 - 15.	For Purity of Boiled Linseed Oil from North American Seed.
D 13 - 24.	For Turpentine.
D 125 - 23.	For Perilla Oil, Raw or Refined.
D 79 - 24.	For Zinc Oxide.
D 80 - 24.	For Leaded Zinc Oxide.
D 81 - 24.	For Basic Carbonate White Lead.
D 82 - 24.	For Basic Sulfate White Lead.
D 83 - 24.	For Red Lead.
D 85 - 24.	For Ocher.

STANDARD METHODS

D 153 - 24.	Of Test for Specific Gravity of Pigments.
D 154 - 24.	Of Testing Oleo-Resinous Varnishes.
D 154 - 25.	Of Testing Oleo-Resinous Varnishes.[1]
D 29 - 24.	Of Testing Shellac.
D 29 - 25.	Of Test for Determination of Wax in Shellac.[2]
*D 56 - 21.	Of Test for Flash Point of Volatile Flammable Liquids.
*D 34 - 17.	Of Routine Analysis of White Pigments.
*D 49 - 18.	Of Routine Analysis of Dry Red Lead.

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* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

[1] To be added to the Standard Methods of Testing Oleo-Resinous Varnishes (Serial Designation: D 154 - 24) when the Book of Standards is next published.

[2] To be added to the present Standard Methods of Testing Shellac (Serial Designation: D 29 - 24) when the Book of Standards is next published.

*D 50 – 18. Of Routine Analysis of Yellow, Orange, Red and Brown Pigments Containing Iron and Manganese.
D 126 – 23. Of Routine Analysis of Yellow and Orange Pigments Containing Chromium Compounds, Blue Pigments and Chrome Green.
D 186 – 25. Of Routine Analysis of Titanium Pigments.

Standard Definitions

D 16 – 24. Of Terms Relating to Paint Specifications.

PETROLEUM PRODUCTS AND LUBRICANTS

Standard Methods

D 47 – 24. Of Test for Specific Gravity of Lubricants.
D 206 – 25. Abridged Volume Correction Table for Petroleum Oils.
D 87 – 22. Of Test for Melting Point of Paraffin Wax.
D 92 – 24. Of Test for Flash and Fire Points by Means of Open Cup.
D 93 – 22. Of Test for Flash Point by Means of the Pensky-Martens Closed Tester.
D 95 – 24. Of Test for Water in Petroleum Products and Other Bituminous Materials.
D 96 – 24. Of Test for Water and Sediment in Petroleum Products by Means of Centrifuge.

ROAD MATERIALS

Standard Specifications

*D 57 – 20. For Materials for Cement Grout Filler for Brick and Stone Block Pavements.
D 58 – 24. For Materials for Cement Mortar Bed for Brick, Stone Block, Wood Block, Asphalt Block and Other Block Pavements.
*D 59 – 22. For Block for Granite Block Pavements.
D 131 – 23. For Block for Recut Granite Block Pavements.
D 132 – 23. For Block for Durax Granite Pavements.
D 65 – 23. For Broken Slag for Waterbound Base and Wearing Course.
D 66 – 23. For Shovel-Run or Crusher-Run Broken Slag for Waterbound Base.

Standard Methods

D 2 – 08. Of Test for Abrasion of Road Material.
*D 3 – 18. Of Test for Toughness of Rock.
*D 30 – 18. Of Test for Apparent Specific Gravity of Course Aggregates.
D 55 – 25. Of Test for Apparent Specific Gravity of Sand, Stone and Slag Screenings, and Other Fine Non-Bituminous Highway Materials.
D 72 – 21. Of Test for Quantity of Clay and Silt in Gravel for Highway Construction.
*D 75 – 22. Of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods.

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* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

D 7 – 18. Of Mechanical Analysis of Sand or Other Fine Highway Material, except Fine Aggregates Used in Cement Concrete.
D 18 – 16. Of Mechanical Analysis of Broken Stone or Broken Slag, except Aggregates Used in Cement Concrete.
D 19 – 16. Of Mechanical Analysis of Mixtures of Sand or Other Fine Material with Broken Stone or Broken Slag, except Aggregates Used in Cement Concrete.
D 35 – 18. Form of Specifications for Certain Commercial Grades of Broken Stone.
D 5 – 25. Of Test for Penetration of Bituminous Materials.
D 6 – 20. Of Test for Loss on Heating of Oil and Asphaltic Compounds.
*D 20 – 18. Of Test for Distillation of Bituminous Materials Suitable for Road Treatment.
D 36 – 24. Of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method).
D 61 – 24. Of Test for Softening Point of Tar Products (Cube-in-Water Method).
D 140 – 25. Of Sampling Bituminous Materials.

STANDARD DEFINITIONS

D 8 – 18. Of Terms Relating to Materials for Roads and Pavements.

COAL AND COKE

STANDARD SPECIFICATIONS

D 17 – 16. For Foundry Coke.
D 166 – 24. For Gas and Coking Coals.

STANDARD METHODS

*D 21 – 16. Of Sampling Coal.
D 22 – 24. Of Laboratory Sampling and Analysis of Coal.
D 37 – 24. Of Laboratory Sampling and Analysis of Coke.
D 141 – 23. Of Shatter Test for Coke.
D 167 – 24. Of Test for Volume of Cell Space of Lump Coke.

TIMBER AND TIMBER PRESERVATIVES

STANDARD SPECIFICATIONS

D 10 – 15. For Yellow-Pine Bridge and Trestle Timbers.
D 24 – 20. For Southern Yellow-Pine Timber to be Creosoted.
D 25 – 20. For Southern Yellow-Pine Piles and Poles to be Creosoted.
D 52 – 20. For Wooden Paving Blocks for Exposed Pavements.

STANDARD METHODS

D 38 – 24. Of Sampling and Analysis of Creosote Oil.

STANDARD DEFINITIONS

D 9 – 15. Of Terms Relating to Structural Timber.

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* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

WATERPROOFING

STANDARD SPECIFICATIONS

D 40 – 25. For Asphalt for Use in Damp-proofing and Waterproofing Below Ground Level.
D 144 – 25. For Asphalt for Use in Damp-proofing and Waterproofing Above Ground Level.
D 41 – 25. For Primer for Use with Asphalt in Damp-proofing and Waterproofing Below and Above Ground Level.
D 42 – 25. For High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level.
D 145 - 25. For High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level.
D 200 – 25. For High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level.
D 201 – 25. For High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level.
D 43 – 25. For Creosote Oil for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing Below and Above Ground Level.
D 169 – 25. For Ashpalt Mastic for Use in Waterproofing.
D 170 – 25. For Bituminous Grout for Use in Waterproofing Above Ground Level.
D 171 – 25. For Bituminous Grout for Use in Waterproofing Below Ground Level.
D 173 – 25. For Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing.
D 174 – 25. For Burlap Saturated with Bituminous Substances for Use in Waterproofing.

INSULATING MATERIALS

STANDARD METHODS

D 48 – 24. Of Testing Molded Insulating Materials.
D 117 – 24. Of Testing Transformer and Switch Oils.

RUBBER PRODUCTS

STANDARD SPECIFICATIONS

D 14 – 23. For Cotton Rubber-Lined Fire Hose for Private Fire Department Use.
D 26 – 23. For Cotton Rubber-Lined Fire Hose for Public Fire Department Use.
D 46 – 24. For Wrapped Air Hose for Use with Pneumatic Tools.
D 60 – 24. For Braided Air Hose for Use with Pneumatic Tools.
D 177 – 24. For Wrapped Cold Water Hose.
D 53 – 24. For Rubber Belting for Power Transmission.
D 120 – 23. For Rubber Gloves for Electric Workers on Apparatus or Circuits Not Exceeding 3000 Volts to Ground.

D 178 – 24. For Rubber Matting for Use Around Electrical Apparatus or Circuits Not Exceeding 3000 Volts to Ground.
D 151 – 23. For Rubber Pump Valves.
D 69 – 24. For Adhesive Tape for General Use for Electrical Purposes.

Standard Methods

D 15 – 24. Of Testing Rubber Products.

TEXTILE MATERIALS

Standard Specifications

D 122 – 25. For Imperfections and Tolerances for Square-Woven Tire Fabrics.
D 179 – 25. For Imperfections and Tolerances for Cord Tire Fabrics.
D 181 – 25. For Tolerances for Hose Ducks and Belt Ducks.
D 203 – 25. For Tolerances and Test Methods for Electrical Cotton Yarns.
D 205 – 25. Specifications and Tests for Osnaburg Cement Sacks.

Standard Methods

D 39 – 24. Of Testing Cotton Fabrics.

THERMOMETERS

Standard Specifications

D 182 – 25. For A.S.T.M. Partial-Immersion Thermometer for General Use, −20 to +150° C., 0 to +300° F.
D 183 – 25. For A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +300° C., +20 to +580° F.
D 184 – 25. For A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +400° C., +20 to +760° F.

E. MISCELLANEOUS SUBJECTS

Standard Methods

E 4 – 24. Of Verification of Testing Machines.
E 3 – 24. Of Metallographic Testing of Iron and Steel.
E 5 – 24. Of Metallographic Testing of Non-Ferrous Metals and Alloys.

Standard Rules

E 2 – 24. Governing the Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography.

Standard Definitions

E 7 – 24. Of Terms Relating to Metallography.

LIST OF TENTATIVE STANDARDS[1]

Published Collectively in their latest revised form in
1925 BOOK OF A.S.T.M. TENTATIVE STANDARDS

Those Tentative Standards bearing the final designation of any given year are published in the annual Proceedings, Part I, of that year. Thus, Tentative Standards having the final designation "25 T" appear in this volume (see Table of Contents); those having the final designation "24 T" appear in Proceedings, Vol. 24, Part I; etc.

The term "Tentative Standard" is applied to a proposed Standard which is printed in the Proceedings for one or more years with a view of eliciting criticism, of which the committee concerned will take due cognizance before recommending final action towards the adoption of such Tentative Standard by formal action of the Society.

These Tentative Standards are all copyrighted in the name of the American Society for Testing Materials. Permission to reprint any of them can be obtained only from the Executive Committee on application to the Secretary-Treasurer.

In the serial designations prefixed to the following titles, the initial letter indicative of the general classification and the first number are permanent, and are retained when a Tentative Standard is adopted as Standard. The final number indicates the year of original issue or, in the case of revision, the year of last revision. In using Tentative Standards, care should be taken to refer to the latest revision which may always be found in the Book of A.S.T.M. Tentative Standards issued yearly about October 1. The letter "T" is appended to the designation of all Tentative Standards. The serial designations of Tentative Standards which have for any reason not been adopted as standard and have been discontinued, are permanently dropped. In ordering Tentative Standards, the complete serial designations should be stated, thus, A 67 – 20 T, etc.

A. FERROUS METALS

STEEL

TENTATIVE SPECIFICATIONS

A 67 – 20 T.	For Steel Tie Plates.
A 71 – 24 T.	For Carbon Tool Steel.
A 92 – 24 T.	For High-Speed Tool Steel.
A 76 – 20 T.	For Low-Carbon Steel Track Bolts.
A 77 – 20 T.	For Electric Cast-Steel Anchor Chain.
A 78 – 23 T.	For Steel Plates of Structural Quality for Forge Welding.
A 89 – 23 T.	For Steel Plates of Flange Quality for Forge Welding.
A 82 – 21 T.	For Cold-Drawn Steel Wire for Concrete Reinforcement.
A 87 – 22 T.	For Carbon-Steel Castings for Railroads.
A 21 – 25 T.	For Carbon-Steel Car and Tender Axles.[2]

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[1] As of August 28, 1925. Total number, 193.

[2] To replace, when adopted, the present Standard Specifications for Carbon-Steel Car and Tender Axles (Serial Designation: A 21 – 18).

A 94 – 25 T.	For Structural Silicon Steel.
A 95 – 25 T.	For Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service.
A 96 – 25 T.	For Alloy-Steel Bolting Material for High-Temperature Service.
A 93 – 24 T.	For Hot-Dipped Galvanized Sheets.

TENTATIVE RECOMMENDED PRACTICE

A 37 – 25 T.	For Carburizing and Heat Treatment of Carburized Objects.[1]

FERRO-ALLOYS

TENTATIVE SPECIFICATIONS

A 97 – 25 T.	For Tungsten Powder.
A 98 – 25 T.	For Spiegeleisen.
A 99 – 25 T.	For Ferro-Maganese.
A 100 – 25 T.	For Ferro-Silicon.
A 101 – 25 T.	For Ferro-Chromium.
A 102 – 25 T.	For Ferro-Vanadium.

TENTATIVE METHODS

A 103 – 25 T.	Of Sampling Ferro-Alloys.
A 104 – 25 T.	Of Chemical Analysis of Ferro-Alloys.

WROUGHT IRON

TENTATIVE SPECIFICATIONS

A 86 – 25 T.	For Hollow Staybolt Iron.

B. NON-FERROUS METALS

TENTATIVE SPECIFICATIONS

B 51 – 24 T.	For Phosphor Tin.
B 52 – 24 T.	For Phosphor Copper.
B 53 – 24 T.	For Silicon Copper.
B 26 – 25 T.	For Aluminum Base Alloy Sand Castings.
B 9 – 25 T.	For Bronze Trolley Wire.[2]
B 48 – 23 T.	For Soft Rectangular Copper Wire.
B 49 – 23 T.	For Hot-Rolled Copper Rods for Wire Drawing.
B 7 – 24 T.	For Manganese-Bronze Ingots for Sand Castings.
B 54 – 24 T.	For Manganese-Bronze Sand Castings.
B 57 – 25 T.	For Muntz Metal Condenser Tube Plates.
B 17 – 21 T.	For Non-Ferrous Alloys for Railway Equipment in Ingots, Castings, and Finished Car and Tender Bearings.
B 23 – 18 T.	For White Metal Bearing Alloys (known commercially as "Babbitt Metal").
B 24 – 25 T.	For Aluminum Ingots for Remelting.
B 25 – 19 T.	For Aluminum Sheet.
B 37 – 20 T.	For Aluminum for Use in the Manufacture of Iron and Steel.
B 50 – 25 T.	For Non-Ferrous Insect Screen Cloth.

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[1] To replace, when adopted, the present Recommended Practice for Heat Treatment of Case-Hardened Carbon-Steel Objects (Serial Designation: A 37 – 14).

[2] To replace, when adopted, the present Standard Specifications for High-Strength Bronze Trolley Wire, Round and Grooved: 40 and 65-per-cent Conductivity (Serial Designation: B 9 – 16).

TENTATIVE METHODS

B 40 – 25 T.	Of Chemical Analysis of Aluminum and Light Aluminum Alloys.

C. CEMENT, LIME, GYPSUM, AND CLAY PRODUCTS

TENTATIVE SPECIFICATIONS

C 9 – 16 T.	Specifications and Tests for Compressive Strength of Portland-Cement Mortars.[1]
C 15 – 17 T.	For Required Safe Crushing Strengths of Sewer Pipe to Carry Loads from Ditch Filling.
C 5 – 24 T.	For Quicklime for Structural Purposes.
C 46 – 25 T.	For Quicklime for Use in the Manufacture of Sulfite Pulp.
C 47 – 22 T.	For Hydrated Lime for the Manufacture of Varnish.
C 53 – 25 T.	For Quicklime for Use in Water Treatment.
C 54 – 25 T.	For Hydrated Lime for Use in Water Treatment.
C 33 – 23 T.	For Concrete Aggregates.
C 55 – 24 T.	For Concrete Building Brick.
C 34 – 24 T.	Specifications and Tests for Hollow Burned-Clay Load-Bearing Wall Tile.
C 56 – 24 T.	Specifications and Tests for Hollow Burned-Clay Fireproofing, Partition and Furring Tile.
C 57 – 24 T.	Specifications and Tests for Hollow Burned-Clay Floor Tile.

TENTATIVE METHODS

C 25 – 25 T.	Of Chemical Analysis of Limestone, Quicklime and Hydrated Lime.
C 50 – 24 T.	Of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products.
C 38 – 21 T.	Of Test for Resistance of Fire-Clay Brick to Spalling Action.
C 42 – 25 T.	Of Securing Specimens of Hardened Concrete from the Structure.

TENTATIVE DEFINITIONS

C 51 – 24 T.	Of Terms Relating to Lime.
C 11 – 25 T.	Of Terms Relating to the Gypsum Industry.
C 58 – 25 T.	Of the Term Sand.

TENTATIVE RULES

C 44 – 22 T.	For Inspection of Concrete and Reinforced Concrete Work.

D. MISCELLANEOUS MATERIALS

PRESERVATIVE COATINGS

TENTATIVE SPECIFICATIONS

D 12 – 25 T.	For Raw Tung Oil.
D 124 – 22 T.	For Soya Bean Oil, Raw or Refined.
D 51 – 18 T.	For Foots Permissible in Properly Clarified Pure Raw Linseed Oil from North American Seed.
D 77 – 21 T.	For Purity of Raw Linseed Oil from South American Seed.

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[1] To be added, when adopted, to the Standard Specifications and Tests for Portland Cement (Serial Designation: **C 9 – 21**).

D 78 - 21 T.	For Purity of Boiled Linseed Oil from South American Seed.
D 207 - 25 T.	For Dry Bleached Shellac.
D 84 - 25 T.	For Iron Oxide and Iron Hydroxide.
D 208 - 25 T.	For Lithopone.
D 209 - 25 T.	For Lampblack.
D 210 - 25 T.	For Bone Black
D 211 - 25 T.	For Chrome Yellow.
D 212 - 25 T.	For Pure Chrome Green.
D 213 - 25 T.	For Reduced Chrome Green.

TENTATIVE METHODS

D 214 - 25 T.	Of Testing Shellac Varnish.
D 185 - 24 T.	Of Test for Coarse Particles in Paint Pigments.
D 215 - 25 T.	Of Routine Analysis of White Linseed Oil Paints.

PETROLEUM PRODUCTS AND LUBRICANTS

TENTATIVE METHODS

D 86 - 25 T.	Of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products.
D 216 - 25 T.	Of Test for Distillation of Natural Gas Gasoline.
D 127 - 24 T.	Of Test for Melting Point of Petrolatum.
D 128 - 24 T.	Of Analysis of Grease.
D 217 - 25 T.	Of Test for Penetration of Greases.
D 88 - 25 T.	Of Test for Viscosity of Petroleum Products and Lubricants.
D 155 - 23 T.	Of Test for Color of Lubricating Oils by Means of Union Colorimeter.
D 218 - 25 T.	Of Test for Color of Petrolatum by Means of the Union Colorimeter.
D 156 - 23 T.	Of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer.
D 187 - 24 T.	Of Test for Burning Quality of Kerosine Oils.
D 219 - 25 T.	Of Test for Burning Quality of Long-Time Burning Oil for Railway Use.
D 90 - 24 T.	Of Test for Sulfur in Naphthas and Illuminating Oils.
D 129 - 22 T.	Of Test for Sulfur in Petroleum Oils Heavier than Illuminating Oil.
D 130 - 22 T.	Of Test for Detection of Free Sulfur and Corrosive Sulfur Compounds in Gasoline.
D 91 - 21 T.	Of Test for Precipitation Number of Lubricating Oils.
D 157 - 23 T.	Of Test for Steam Emulsion of Lubricating Oils.
D 94 - 21 T.	Of Test for Saponification Number.
D 188 - 25 T.	Of Test for Neutralization Number of Petroleum Products and Lubricants.
D 189 - 24 T.	Of Test for Carbon Residue of Lubricants.
D 97 - 25 T.	Of Test for Cloud and Pour Points of Petroleum Products.
D 158 - 25 T.	Of Testing Gas Oils (Gravity, Distillation, Sulfur, Carbon Residue, Pour Point, Viscosity, Water).

ROAD MATERIALS

TENTATIVE SPECIFICATIONS

D 63 - 23 T. For Commercial Sizes of Broken Stone and Broken Slag for Highway Construction.
D 64 - 20 T. For Commercial Sizes of Sand and Gravel for Highway Construction.
D 190 - 24 T. For Broken Stone for Waterbound Base.
D 191 - 24 T. For Broken Stone for Waterbound Macadam Surface Course.
D 192 - 24 T. For Broken Stone for Bituminous Macadam.
D 193 - 24 T. For Broken Stone for Bituminous Concrete Base.
D 194 - 24 T. For Broken Stone for Bituminous Concrete Surface.
D 195 - 24 T. For Broken Slag for Bituminous Macadam Base.
D 196 - 24 T. For Broken Slag for Bituminous Concrete Base.
D 159 - 24 T. For Broken Slag for Bituminous Macadam Wearing Course.
D 160 - 24 T. For Broken Slag for Bituminous Concrete (Coarse-Graded Aggregate Type).
D 161 - 24 T. For Broken Slag for Bituminous Concrete (Fine-Graded Aggregate Type).
D 162 - 23 T. For Sand for Sheet Asphalt and Bituminous Concrete Pavements.
D 67 - 23 T. For Natural or Artificial Sand-Clay Mixtures for Road Surfacing.
D 98 - 22 T. For Calcium Chloride for Dust Prevention.
D 133 - 23 T. For Asphalt Cement, 10 to 15 Penetration, for the Manufacture of Asphalt Block.
D 134 - 23 T. For Asphalt Cement, 15 to 25 Penetration, for the Manufacture of Asphalt Block.
D 163 - 23 T. For Asphalt Cement, 25 to 30 Penetration, for Use in Sheet Asphalt and Asphaltic Concrete Pavements.
D 164 - 23 T. For Asphalt Cement, 30 to 40 Penetration, for Use in Sheet Asphalt and Asphaltic Concrete Pavements.
D 99 - 23 T. For Asphalt Cement, 40 to 50 Penetration, for Use in Sheet Asphalt and Asphaltic Concrete Pavements.
D 100 - 23 T. For Asphalt Cement, 50 to 60 Penetration, for Use in Sheet Asphalt and Asphaltic Concrete Pavements.
D 101 - 23 T. For Asphalt Cement, 60 to 70 Penetration, for Use in Sheet Asphalt, Asphaltic Concrete, and Asphalt Macadam Pavements.
D 102 - 24 T. For Asphalt Cement, 85 to 100 Penetration, for Use in Asphalt Macadam Pavements.
D 103 - 24 T. For Asphalt Cement, 100 to 120 Penetration, for Use in Asphalt Macadam Pavements.
D 135 - 23 T. For Asphalt Cement, 120 to 150 Penetration, for Use in Asphalt Macadam Pavements.
D 104 - 23 T. For High-Carbon Tar for Surface Treatment, Cold Application.
D 105 - 23 T. For Low-Carbon Tar for Surface Treatment, Cold Application.
D 106 - 25 T. For High-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product).
D 107 - 23 T. For Low-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product).

D 108 - 23 T.	For High-Carbon Tar for Surface Treatment, Hot Application.
D 109 - 23 T.	For Low-Carbon Tar for Surface Treatment, Hot Application.
D 110 - 25 T.	For High-Carbon Tar Cement.
D 111 - 23 T.	For Low-Carbon Tar Cement.
D 112 - 23 T.	For Coal-Tar Pitch for Stone Block Filler.

TENTATIVE METHODS

D 70 - 20 T.	Of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cement and Soft Tar Pitches.
D 71 - 20 T.	Of Test for Specific Gravity of Asphalts and Tar Pitches Sufficiently Solid to be Handled in Fragments.
D 73 - 20 T.	Of Test for Quantity of Clay in Sand-Clay, Topsoil and Semi-Gravel for Highway Construction.
D 136 - 22 T.	Of Decantation Test for Sand and Other Fine Aggregates.
D 137 - 25 T.	Of Mechanical Analysis of Subgrade Soils.
D 220 - 25 T.	Of Test for the Determination of the Moisture Equivalent of Subgrade Soils in the Field.
D 138 - 25 T.	Of Test for Consistency of Portland-Cement Concrete.
D 4 - 23 T.	Of Test for the Determination of Bitumen.
D 165 - 23 T.	Of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride.
D 113 - 22 T.	Of Test for Ductility of Bituminous Materials.
D 139 - 25 T.	Of Float Test for Bituminous Materials.

COAL AND COKE

TENTATIVE METHOD

D 197 - 24 T.	Of Test for Fineness of Powdered Coal.

TENTATIVE DEFINITIONS

D 121 - 24 T.	Of Terms Relating to Coke.
D 142 - 25 T.	Of Terms Relating to Coal.

TIMBER

TENTATIVE SPECIFICATIONS

D 23 - 20 T.	For Structural Douglas Fir.

TENTATIVE METHODS

D 143 - 24 T.	Of Testing Small Clear Specimens of Timber.
D 198 - 24 T.	For Conducting Static Tests of Timber in Structural Sizes.
D 168 - 23 T.	Of Test for Coke Residue of Creosote Oil.[1]
D 199 - 24 T.	Of Chemical Analysis of Zinc Chloride.

SLATE

TENTATIVE METHODS

D 221 - 25 T.	Of Test for Water Absorption of Slate.
D 222 - 25 T.	Of Flexure Testing of Slate.

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[1] **To be added, when adopted, to the present Standard Methods of Sampling and Analysis of Creosote Oil (Serial Designation: D 38 - 24).**

WATERPROOFING

TENTATIVE SPECIFICATIONS

D 223 – 25 T.	For Acid Resisting Asphalt Mastic.
D 224 – 25 T.	For Smooth-Surfaced Asphalt Roll-Roofing.
D 225 – 25 T.	For Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles.
D 226 – 25 T.	For Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs.
D 227 – 25 T.	For Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs.

TENTATIVE METHODS

D 146 – 25 T.	Of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing.
D 147 – 25 T.	Of Testing Bituminous Mastics, Grouts and Like Mixtures.
D 228 – 25 T.	Of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles.

INSULATING MATERIALS

TENTATIVE METHODS

D 115 – 25 T.	Of Testing Insulating Varnishes.
D 116 – 24 T.	Of Testing Electrical Porcelain.
D 149 – 24 T.	Of Testing Sheet and Tape Insulating Materials for Dielectric Strength.
D 150 – 23 T.	Of Testing Electrical Insulating Materials for Phase Difference (Power Factor) and Dielectric Constant at Radio Frequencies.
D 175 – 25 T.	Of Testing Electrical Insulating Materials for Voltage Effects at Radio Frequencies.
D 176 – 25 T.	Of Testing Cable Splicing and Pothead Compounds.
D 202 – 25 T.	Of Testing Untreated Insulating Paper.
D 229 – 25 T.	Of Testing Laminated Sheet Insulating Materials.

SHIPPING CONTAINERS

TENTATIVE SPECIFICATIONS

D 44 – 20 T.	For Wooden Boxes, Nailed and Lock-Corner Construction, for the Shipment of Canned Foods.
D 45 – 17 T.	For Canned Foods Boxes, Wirebound Construction.
D 68 – 22 T.	For Wooden Boxes, Nailed and Lock-Corner Construction.
D 118 – 21 T.	For 4-One Boxes and Similar Type Boxes.

RUBBER PRODUCTS

TENTATIVE SPECIFICATIONS

D 27 – 21 T.	For Insulated Wire and Cable: 30-per-cent Hevea Rubber.
D 54 – 24 T.	For Steam Hose.
D 119 – 22 T.	For Rubber Insulating Tape.

TEXTILE MATERIALS

TENTATIVE SPECIFICATIONS

D 76 - 25 T.	For Textile Testing Machines.
D 180 - 25 T.	For Tolerances and Test Methods for Cotton Yarns, Single and Plied.
D 204 - 25 T.	For Tolerances and Test Methods for Cotton Sewing Threads.
D 230 - 25 T.	For Tolerances for Numbered Cotton Duck.
D 231 - 25 T.	For Tolerances and Test Methods for Knit Goods.

TENTATIVE METHODS

D 152 - 22 T.	Of Testing Cotton Fibers.
D 232 - 25 T.	Of Testing Grease Wool and Allied Fibers for Scoured Content.

TENTATIVE DEFINITIONS

D 123 - 22 T.	Of Terms Relating to Textile Materials.

E. MISCELLANEOUS SUBJECTS

TENTATIVE SPECIFICATIONS

E 11 - 25 T.	For Sieves for Testing Purposes.

TENTATIVE METHODS

E 8 - 25 T.	Of Tension Testing of Metallic Materials.
E 9 - 25 T.	Of Compression Testing of Metallic Materials.
E 10 - 25 T.	Of Brinell Hardness Testing of Metallic Materials.
E 4 - 24 T.	Of Verification of Testing Machines by Means of an Elastic Calibration Bar.[1]

TENTATIVE DEFINITIONS

E 6 - 25 T.	Of Terms Relating to Methods of Testing.
E 12 - 25 T.	Of Terms Relating to Specific Gravity.
E 13 - 25 T.	Of the Term Screen (Sieve).

TENTATIVE RECOMMENDED PRACTICE

E 14 - 25 T.	For Thermal Analysis of Steel.

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[1] To be added, when adopted, to the present Standard Methods of Verification of Testing Machines (Serial Designation: E 4 - 24).